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UNITED NATIONS CONFERENCE
ON THE APPLICATION OF SCIENCE AND TECHNOLOGY
FOR THE BENEFIT OF THE LESS DEVELOPED AREAS

Agenda Item: D.11.2

Norway

THE ODDA PROCESS AND ITS APPLICATION IN LESS DEVELOPED AREAS

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Papers have been contributed to the United Nations Conference on the Application of Science and Technology for the Benefit of the Less Developed Areas as working papers for that Conference. They are published as presented by the contributors, and the contents and the views expressed are those of the contributors. (See notes overleaf.)

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The Odda Process and its Application in Less Developed Areas.

By L. Torvund, chem. eng., Norsk Hydro, Norway

1. Introduction

The fertilizer industry will be in a key position when modern agriculture and industry are going to be built up in less developed areas. It is not obvious though, that the structure of this industry in the old countries is adequate.

The new countries which in comparatively few years have to increase their production, must examine very carefully which processes to select for development of new fertilizer industry.

The production of ammonia and the supply of rock phosphate are two fundamental constituents for the fertilizer industry. To build a sulphuric acid plant especially for fertilizer production is no longer a necessity.

The production of concentrated complex fertilizers based on nitric acid may often be a preferable alternative. By treatment of rock-phosphate with nitric acid one obtains valuable calcium nitrate as a by-product instead of calcium sulphate from the sulphuric acid process.

The best existing fertilizer process based on the raw materials rock phosphate, nitric acid and ammonia is the Odda-process. Its main product is a nitrogen-phosphorus fertilizer usually with 20 % N and 9 % P., but the N/P ratio may be varied within wide limits. During the final granulation process other plant nutrients may be added in wanted amounts. A by-product of the process is calcium-nitrate, normally converted to prilled nitrate of lime with 15.5 % N.

The Odda process was invented by Erling B. Johnson, Odda, Norway, in 1928 and originally patented in Norwegian p. nr. 48644, and later in other countries, as British p. nr. 339340, French p. nr. 682423 and German p. nr. 573284. Several additional patents are also issued.

The process is used in Germany, Norway, Holland, France and Finland. Between 1.5 and 2.0 million tonnes complex fertilizer were produced last year by this process. - The production capa ity is being increased. In Norway more than half the total fertilizer production, in Germany about a quarter and in Holland and France between 10 and 20 percent is made by the Odda process. - The different firms using the process have gained a considerable amount of experience, and technical problems arising during production have been overcome. The problems to be solved will be limited to local adaptability.

2. Description of the Odda Process

It is outside the scope of this paper to deal with the production of ammonia and nitric acid. It will here be assumed that these products are available from separate plants. It is also assumed that there is access to phosphate rock and other raw materials. Electric energy and cooling water must also be available at the site.

In figure 1. a flow sheet is given for the production of fertilizer by the Odda process. The main steps are as follows: - Phosphate rock is digested in nitric acid and the produced calcium nitrate tetrahydrate is crystallized out by cooling of the digested phosphate solution. The crystals are removed, melted, neutralised with ammonia and the water content reduced by evaporation. Finally nitrate of lime is prilled, cooled and screened. After the removal of the tetrahydrate crystals, the mother liquor is neutralised with ammonia, concentrated by evaporation, granulated - possibly with addition of potassium salts and other plant nutrients -, dryed, cooled and screened.

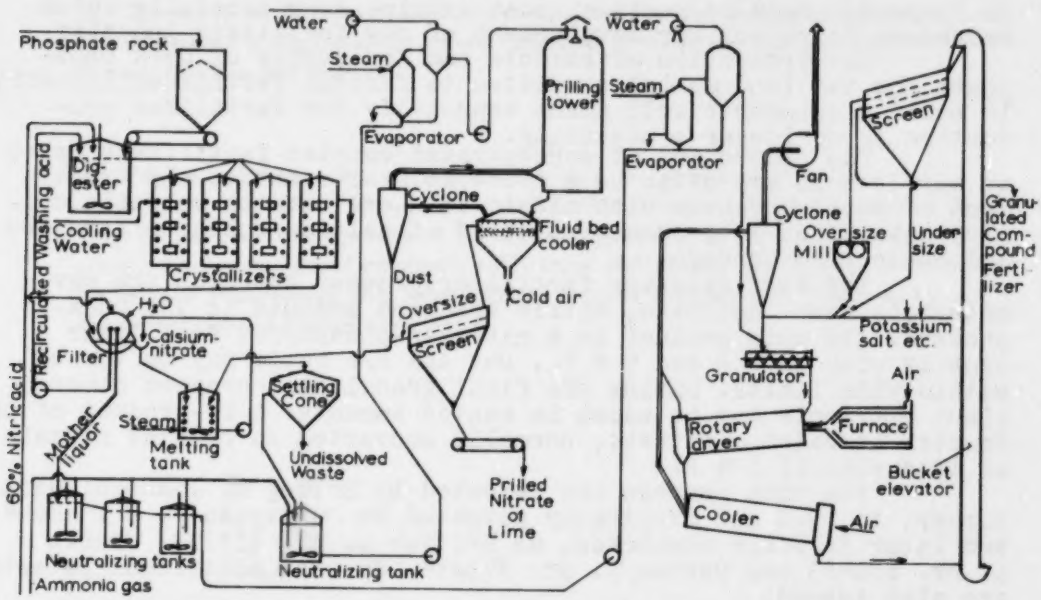

Fig. 1. Production of Granulated Compound Fertilizer and Prilled Nitrate of Lime by the Odda Process.

2 a. Raw Materials

Fundamentally any phosphate rock based on fluorine or hydroxy-apatite may be used. Chlorine-apatite will lead to

prohibitive corrosion problems. For favourable accomplishment of the process some limiting factors should be taken into account.

To obtain effective digestion 90 - 95 % of the ground phosphate should be less than 0.5 millimetres in diametre. Afterwards it should be dry enough for easy mechanical handling. Acid insoluble constituents ought not be more than two percent. A higher content is not prohibitive, but should preferably be separated from the digested liquor. Of acid soluble constituents, the highest possible content of calcium phosphate, $Ca_3(PO_4)_2$, is desirable, preferably not less than 75 %. The best available phosphate rocks contain about 85 %. A certain amount of $CaCO_3$ is by itself not harmful, but it will increase the amount of calcium nitrate to be removed. Iron compounds may cause a reduction of citrate soluble phosphorus in the final product and should preferably not exceed 0.7 - 0.9 %, - calculated as iron. Organic matter may act as foam stabilizers and lead to troublesome foaming. The problem may be overcome either with antifoaming agents or by mechanical foam killing devices.

In general all commercial phosphate rocks on the world market can be used for the process. It should also be possible to adapt the process to more special types if necessary.

The nitric acid should be in the concentration range 58 - 61 %. Ammonia may be used either in the liquid or the gas phase. Ammonia mixed with inert gases may also be used.

2 b. Digestion of Phosphate Rock with Nitric Acid

Ground phosphate rock and nitric acid are continously proportioned in a mixer tank fitted with overflow. In some cases the necessary digestion time makes it desirable to divide the system into several mixing tanks in series.

The proportion of acid to phosphate depends to a certain degree on the type of phosphate used. An excess of at least 15 %, preferably 20 - 25 % of the calculated amount of acid should be used to obtain favourable working conditions. For common types of phosphate rocks 1.3 - 1.5 parts of HNO_3 to 1.0 part of phosphate rock, weight/weight, gives the right ratio.

The exothermic dissolution process yields about 70 M cal per tonne dissolved phosphate rock. The temperature rise is 45°C. In normal conditions the temperature of the digested solution is between 55 - 70°C. The solution consists of a mixture of nitric, phosphoric and a little hydrofluoric acid, as well as dissolved calcium nitrate with different other dissolved and undissolved impurities.

2 c. Crystallization of Calcium Nitrate.

The saturation point of calcium nitrate is obtained in the temperature interval 25 - 28°C when the warm phosphate solution is cooled. By continued cooling $Ca(NO_3)_2 \cdot 4H_2O$ is crystallized out. This is the key point of the Odda process. Crystallization is used for removal of excess, undesired calcium from the phosphate solution. In figure 2 the Ca/P ratio of the mother liquor is plotted

versus the temperature. The graph indicates the amount of $Ca(NO_3)_2 \cdot 4H_2O$ crystallized out.

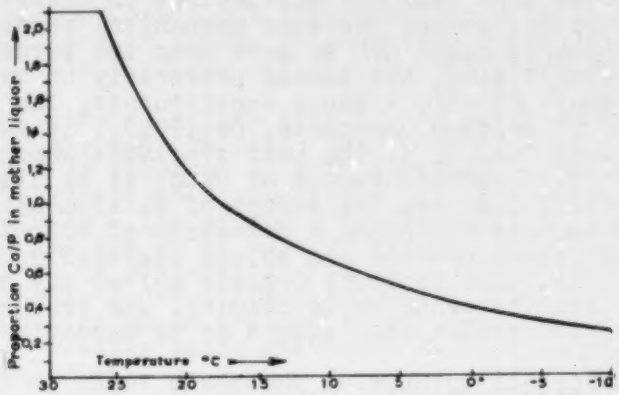

Fig. 2. Crystallization of $Ca(NO_3)_2$ from digested phosphatic liquor of the composition given in the later material balance example.

The cooling process is normally carried out in vessels equipped with stirring and coils cooled by water or circulating brine. The process can be carried out continuously or batchwise. Both systems are used and each have their particular advantages and disadvantages. Continuous crystallization has the advantage of uniform flow which is suitable for the rest of the process. The crystallizers however, have to be shut down at few hours intervals to remove salt deposits from the cooling surface. The method also gives crystals of varied size, and mixed with the undissolved impurities the resulting salt cake will be rather compact making the passage of mother liquor difficult. Effective centrifuges are necessary for the removal of mother liquor from the solid matter.

The batch process has the disadvantage of interrupted flow and requires buffer volume. - In this case though, the regeneration of the cooling surface is a natural part of the batch cycle when filling the crystallizer with warm solution. By seeding at a predetermined supersaturation point it is possible to control the number and the size of the crystals. Very uniform particle size is produced by this method. The crystals form a cake with large open area making the drainage of mother liquor easy. It can be done on a stainless steel mesh filter with a mesh size about 0.5 mm. Small undissolved particles will pass through the cake and filter medium and accompany the mother liquor after filtration.

2 d. Separation of Calcium Nitrate Crystals and Conversion to prilled Nitrate of Lime.

As previously mentioned the separation of the crystals from the mother liquor depends on the crystallisation method used. For continuous crystallization preferably two centrifuges in series

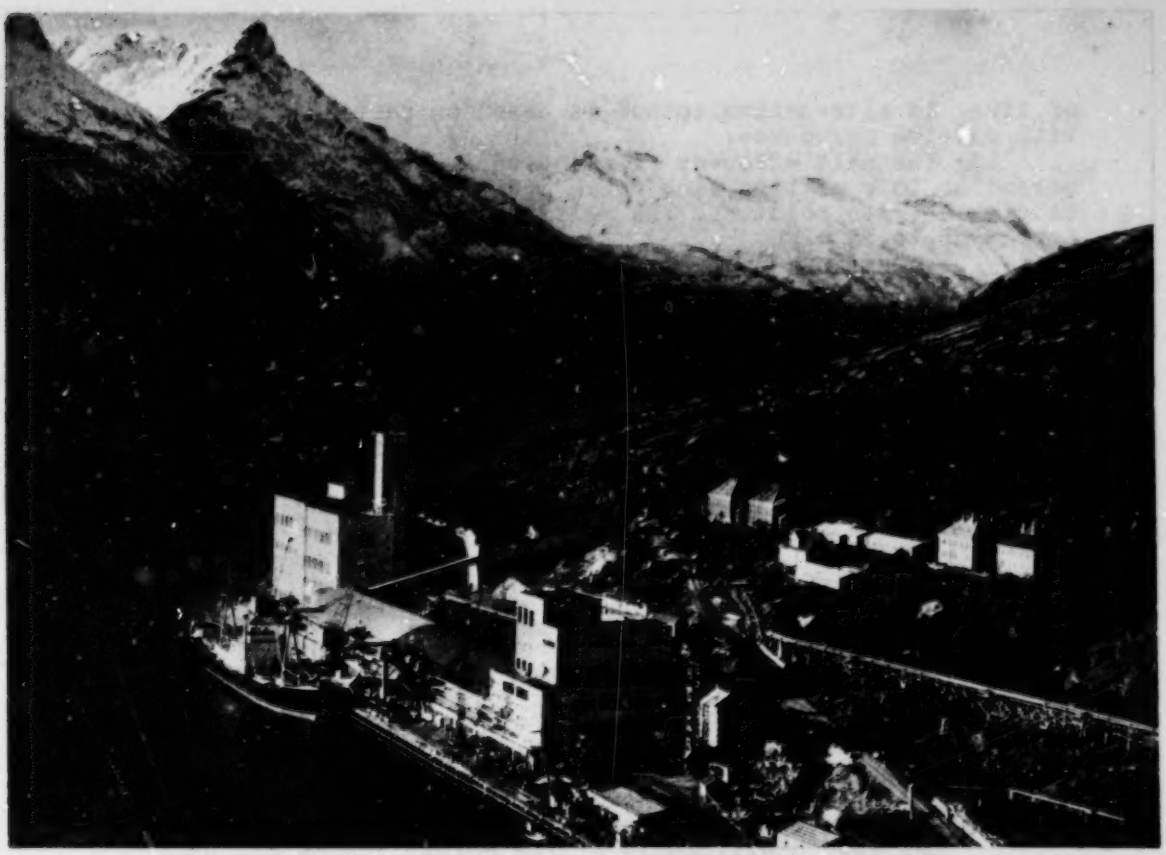

Figure 3.

The Norsk Hydro NPK plant situated 30 kilometres north of the Polar circle at Glomfjord, Norway. About 300 000 tonnes of complex fertilizer is made by the Odda process pro annum.

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are used with an intermediate wash of the crystals in nitric acid. The mixture of nitric acid and mother liquor is returned to the digestion stage.

With batch-crystallization the separation is easier. A continuous vacuum filter with a filter mesh as previously described may be used. The nitric acid wash is carried out on the same filter unit. The capacity is about two tons of dry weight cake per hour per square metre filter area. Washing will reduce the mother liquor content to less than 2 % of cake weight. Some of the nitric acid in the cake may be displaced by water before discharge. In this way it is possible to regulate the ammonium nitrate content in the nitrate

of lime. An alternative method is based on partial neutralization with calcium carbonate.

The salt effluent from the centrifuge or the filter is passed on to a melting tank using a circulating stream of hot calcium nitrate solution. The melting point of calcium nitrate tetrahydrate is 42°C.

The acidic calcium nitrate solution is usually clarified by means of a settling cone, and thereafter neutralized by ammonia gas. The neutral solution is concentrated by evaporation and pumped through spray nozzles into a prilling tower. Here the drops are solidified when falling countercurrently to air with an inlet temperature of 10°C and outlet temperature of about 50°C. The prills from the tower which possess a temperature of about 60°C, are further cooled to room temperature by cold air, preferably in a fluid bed and finally screened. Dust and oversize material is recycled to the melting tank, and the final product of the calcium nitrate stream, prilled nitrate of lime, is ready for storage or packing.

2 e. Neutralization of the Mother Liquor.

The mother liquor from the calcium nitrate separation is passed on to the neutralization stage after adjusting the composition, if necessary, by adding nitric acid and/or phosphate rock.

Neutralization is from a chemical point of view the most complicated step of the process. In spite of considerable research, knowledge of reaction mechanisms and side-reactions is rather limited. The methods and apparatus for carrying out the neutralization are for this reason based mainly upon experience.

The main constituents of the mother liquor are HNO_3, H_3PO_4, HF, H_2SiF_6, $Ca(NO_3)_2$ and H_2O. The main reactions taking place when ammonia is added and the pH raised from 0.5 to 5.5 are the following:

1. $NH_3 + HNO_3 = NH_4NO_3$

2. $2NH_3 + 2HF + Ca(NO_3)_2 = \underline{CaF_2} + 2\ NH_4NO_3$

3. $2NH_3 + H_2SiF_6 + Ca(NO_3)_2 = \underline{CaSiF_6} + 2\ NH_4NO_3$

4. $NH_3 + H_3PO_4 = NH_4H_2PO_4$

5. $NH_3 + NH_4H_2PO_4 = (NH_4)_2HPO_4$

6. $(NH_4)_2HPO_4 + Ca(NO_3)_2 = \underline{CaHPO_4} + 2NH_4NO_3$

Reactions 1, 2 and 3 take place in the pH range 0.5 - 2.5 and the others above pH 2. The main constituents of the neutralized slurry are solid $CaHPO_4$, CaF_2, $CaSiF_6$ and dissolved NH_4NO_3, $NH_4H_2PO_4$ and $(NH_4)_2HPO_4$. The proportion of calcium phosphate to ammonium phosphate will depend upon the Ca/P ratio in the mother liquor. In this way it is possible to regulate the content of water

soluble phosphorus by the removal of calcium nitrate in the crystallization stage.

In addition to the desired main reactions, many others take place. Ammonium calcium phosphate may be formed, and the product may thus contain some (f.ex. 0.5 %) water insoluble nitrogen. Iron content will initiate side-reactions which disturb the phosphate precipitation. Citrate soluble $FePO_4$ as well as more complex citrate insoluble iron phosphates may be formed. Aluminium does not seem to form similar reaction products. Under unfavorable conditions reoccurence of fluorine or hydroxy-apatite may be observed, but this may be avoided by performing the neutralization in several steps.

The heat of neutralization is about 1250 M. cal per tonne NH_3 and will raise the temperature to the boiling point, with considerable evaporation taking place. The viscosity will normally be comparatively low, and the neutralized slurry will easily flow through an evaporator. In certain cases the liquor will have marked thixotropic character, and may become pasty.

Different explanations are given to explain the colloidal conditions which depend on the types of phosphate rock used. The phenomenon is, however, not clearly understood. In specific cases experiments are necessary for the evaluation of rock phosphate to be processed.

Neutralization is normally carried out continuously by flow through two to four succesive vessels equipped with stirring, where the ammonia is put into the liquor. The pH is raised from 1.5 to 3.5 in the first step and to about 5.5 to 6.0 in the last.

The evolution of steam may take place in the reaction vessels, The liquor can also be circulated through separate vacuum evaporators. In the last case the ammonia absorption is better, but the apparatus is more complicated.

The neutralized slurry has a temperature of 115 - 130°C, about 10 - 15 % water and a specific gravity of 1.6. A further reduction of the water content is advantageous before granulation and is normally carried out in vertical tube evaporators at reduced or atmospheric pressure. A water content of about 5 % in the slurry is easily obtained with good flow properties in the temperature range 125 - 140°C.

2 f. Granulation and Drying.

No special modifications are needed for the granulation in the Odda process. Solidification on a water cooled rotary drum followed by drying, screening etc. has been used, but gives a rather flaked and edgy product. Cooling prior to drying is also bad from a heat economic point of view. Prilling in spray towers gives uniform, nice prills for certain qualities, especially for NP-fertilizers when larger amounts of additives(potassium and magnesium salts etc.) are used. The spraying itself, however, is more difficult, and the prilling procedure is not often used for this type of fertilizer .

The method most commonly in use is the following: - Measured quantities of NP-slurry are continuously mixed with weighed amounts of the dry raw materials (potassium and magnesium compounds etc.) in a double screw granulator. Recycled dust and ground oversize product is part of the feed-stock. Moist granules of variable size formed are passed into a direct rotary dryer, usually oilfired. In the first section of the rotary dryer, the granulation is

completed simultaneously with the preliminary drying. Some grinding action takes place to a certain degree in the final drying section and in the following countercurrent cooler. The cooled material is screened; undersize particles below 1.0 - 1.5 mm and oversize product above 3.5 - 4.5 mm is recycled to the granulator, the coarse fraction via a grinder. The exhaust air from the dryer and the cooler is passed through a dust separator and the dust returned to the granulator.

The granulator temperature is 70 - 90°C, outlet temperature from the dryer 90 - 100°C and discharge temperature from the cooler 40 - 80°C.

Exothermic decomposition starts if the material is heated above 200 - 250°C with evolution of nitrous and nitric oxide, nitrogen and ammonia. Nitrosylchloride and chlorine may also be present in the decomposition products. The velocity of the decomposition reaction is rather low, and if discovered on an early stage, reacting lumps can easily be removed. In a later phase the decomposition reaction can be distinguished with water. Because of the decomposition risk, the temperature of the flue gas entering the dryer should not exceed 250°C. For the same reason parallel flow is chosen..

The water content in the material after granulation is 2 - 4 % and in the dryer 0.2 - 1.0 %.

The granulation method normally yields 35 - 45 % product and 55 - 65 % recycled material. Total cycling time is about 50 minutes, of which 25 minutes in the dryer, 20 minutes in the cooler and 5 minutes for the rest.

During storage the fertilizer product may have a tendency for caking. To avoid this, the granulated product is usually powdered with kieselguhr or other suitable conditioning agents.

3. Feedstock, Energy, Investment and Staff Demands.

The production of NP-fertilizer with 20 % N, 9 % P and nitrate of lime with 15.5 % N is chosen in table 1 as a representative exemple. The raw materials are calcined Morocco-phosphate with 16.0 % P and 39.0 % Ca, 60 % nitric acid and ammonia gas.

Table 1

Material balance. Basis 1000 kg P:

	Total amount	NH_3-N	NO_3-N	P	Ca	H_2O
Digestion:	kg	kg	kg	kg	kg	kg
Phosphate rock:	6250	-	-	1000	2440	-
Nitric acid:	14600	-	1945	-	-	5850
Recycled mother liquor:	4000	-	307	312	332	1060
Digested liquor:	24850	-	2252	1312	2772	6910
Crystallization and filtration:						
Removed $Ca(NO_3)_2 \cdot 4H_2O$:	8200	-	973	10	1390	2500
Recycled mother liquor:	4000	-	307	312	332	1060
Mother liquor:	12650	-	972	990	1050	3350
Neutralization:						
Ammonia gas:	1490	1230	-	-	-	-
Water evaporated by heat of reaction:	2150	-	-	-	-	2150
Neutralized NP-slurry:	11990	1230	972	990	1050	1200
Evaporation and drying:						
Water removed:	1150	-	-	-	-	1150
NP-fertilizer:	10840	1230	972	990	1030	50

Table 1 continued

Production of nitrate of lime:	Total amount kg	NH_3-N kg	NO_3-N kg	P kg	Ca kg	H_2O kg
Removed $Ca(NO_3)_2 \cdot 4H_2O$:	8200	-	973	10	1390	2500
Remaining water and acid in cake:	770	-	76	-	-	430
Ammonia for neutralization:	92	76	-	-	-	-
Water removed by evaporation:	1882	-	-	-	-	1882
Prilled nitrate of lime:	7180	76	1049	10	1390	1048

The production losses are not taken into account in this balance, but they amount to about 0.5 % of the NO_3-N, to 1.5 - 2.0 % of NH_3-N and 1 % of the phosphorus. The corresponding energy demands for the material balance amounts to:

840 M. calories for crystallization cooling equal to 50 m^3 water inlet temperature 8°C, outlet temperature 25°C. For prilling and cooling the nitrate of lime 18 000 m^3 air at + 10°C and 7000 m^3 air at - 10°C are needed. For an outdoor air temperature of 35°C the energy demand equals 240 M. cal. Nitrogen originally present as liquid ammonia gives 1200 M. cal. of cooling, corresponding to a net excess of 120 M. cal. In addition about 170 m^3 water preferably not warmer than 25°C is needed for condensors etc.

Steam demands are 3.2 ton s for melting, heating and evaporation of nitrate of lime, 0.6 ton s for evaporation of NP-slurry and 1.2 ton s for other purposes, a total of 5.0 ton s. The production of nitric acid from ammonia gives 8.7 tonnes of steam or an over all excess of 3.7 ton s available for other purposes.

The consumption of fuel oil for drying of the NP or NPK fertilizer is about 0.1 ton . The electric energy demand for mechanical power is about 0.7 MWh.

If one assumes that the above data for materials and energy correspond to one hour's production, then the annual production capacity is 85 000 tonnes NP-fertilizer, or f.ex. 130 000 tonnes NPK 13-6-17, - a plant of reasonable size.

The investment cost depends to some extent on local conditions, as a rough guide 2.5 - 3.0 million $. A corresponding nitric acid plant amounts to 1.5 - 2.0 million $. Costs for roads, quays, stores packing equipment etc. come in addition.

In charge of the work a plant superintendant and a plant manager are needed. The necessary additional staff for continuous shift work will be 6 process workers, 1 in the laboratory and 1 foreman. For day time work 4 - 5 general workers with a foreman, 2 - 3 chemists and 4 - 5 skilled maintenance workers with a qualified foreman should be sufficient. It is assumed that more specialized technical services are available outside the works.

4. The Adaptability of the Process to different Regions.

The Odda process as described above, may be applied anywhere in the world assuming the conditions are suitable for making fertilizer, or if desirable, may be modified to suit different demands.

The chemical composition may be varied to meet any agricultural requirement. The proportion of water soluble P to the total available amount, which is normally 1 : 3, may be increased to about 9 : 10 by further cooling in the crystallization stage. A higher concentration of plant nutrients is hereby obtained, corresponding to a maximum of f. ex. 24 % N and 10.5 % P.

The weight ratio N/P should not be lower than 1.8/1 for a normal Ca/P ratio. The content of N may be varied at convenicence upwards. Other plant nutrients may be added to the NP-component.

The production of compound fertilizer may be combined with the production of ammonium phosphate or a mixture of ammonium-phosphate and ammoniumnitrate if a concentrated, entirely water soluble NP fertilizer is desired. (Norwegian p. nr. 86 066, British p. nr. 757 835, French p. nr. 1114 320 and German p. nr. 953 531).

As seen previously, the cooling demand is satisfied by evaporation of input ammonia, but if this cooling capacity should be desirable for other purposes and cooling water of maximum 15°C is available, the Hydro process may be preferable. In this case nitric acid of about 65 % is used and to some extent neutralized by ammonia prior to digestion. By cooling to 23°C sufficient calcium nitrate is removed as $NH_4NO_3 \cdot 5Ca(NO_3)_2 \cdot 10H_2O$. (Norwegian p. nr. 60 374, British p. nr. 391 328, French p. nr. 738 210, German p. nr. 650 344).

In tropic areas the rather hygroscopic nitrate of lime may be found less convenient. In that case it is possible to convert the calcium nitrate into ammonium nitrate limestone by means of NH_3 and CO_2. Another and probably more suitable alternative for tropic areas is to make a nitrogen solution consisting of 34 % calcium nitrate and 32 % ammonium nitrate which has a nitrogen content of 17 % and a crystallization point of 4°C. When the pH of this solution is kept above 7, it can be stored and distributed in mild steel tanks. This application of the separated calcium nitrate will eliminate the nitrate of lime part of the plant, and thus reduce the necessary investment.

Also the NP or NPK fertilizer can be stored and transported in bulk and distributed in containers if that is found convenient.

From an agricultural point of view the NP or NPK fertilizer made by the Odda process with its wide possibilities for variation in composition, should be fit for all main purposes. Up to this time, however, the production and consumption of this fertilizer is mainly concentrated in the northern and the middle part of Europe.

As a rough guidance the fertilizing efficiency corresponds to the same amount of plant nutrients added as straight materials. In some one year field tests a few percent lower effect is found. than in the reference straight material test where superphosphate is used as the phosphorous compound. It is assumed that the lower effect is due to a slower action of the dicalcium phosphate in the compound fertilizer than of the monocalcium phosphate in the superphosphate.

The calcium phosphate not taken up by the plants, will build up a phosphorous surplus in the soil. In the long run it must be assumed that the difference will be negliable. The secondary elements may also be of appreciable importance in these comparative tests.

In more developed countries there is an increasing demand for concentrated ready - to - use fertilizers in order to save costs and labour. This demand will also be present in less developed areas, in any case after some years of development.

The danger for mistakes in the use of fertilizers will be least when the used material has a composition particularly suitable to local conditions.

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By L. TORVUND,
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The Odda Process and its Application in less developed Areas.

By L. Torvund, chem. eng., Norsk Hydro, Norway.

SUMMARY.

When building up a fertilizer industry in less developed areas, products and processes must be chosen by careful evaluation of possibilities and existing trends in this field. In many cases concentrated complex fertilizers will be preferable. Sulfuric acid is not necessary for this production. The acid function in phosphate digestion can profitably be performed by nitric acid, giving valuable calcium nitrate as a byproduct instead of worthless gypsum from the sulfuric acid.

The best process based on nitric acid digestion of phosphate rock is the Odda process, invented in 1928 by Erling Johnson, Odda, Norway. It is used in Germany, Norway, the Netherlands, France and Finland. The production based on this process was last year 1.5 - 2.0 million tonnes of complex fertilizer, and is increasing.

All main types of phosphate rock may be used as raw material, but for the most favorable accomplishment of the process, some limitating specifications as to mechanical properties and composition should be taken into account.

Other raw materials are 60 % nitric acid, gaseous ammonia and possibly other supplementary materials giving the fertilizer the desired content of potassium and secondary minor plant nutrients.

The phosphate rock is digested in an excess of 15 - 25 % nitric acid. The heat of reaction raises the temperature to 55 - 70°C, and the digested phosphatic solution is cooled in crystallizers to 17°C or lower. $Ca(NO_3)_2 \cdot 4H_2O$ will then crystallize, and is removed by centrifuging or filtration. The crystals having a melting point of 42°C are melted, neutralized with ammonia, concentrated by evaporation and prilled in a spray tower as nitrate of lime. In tropic areas the hygroscopic nitrate of lime is somewhat inconvenient to handle, and in this case the calcium nitrate may be converted to ammonium nitrate limestone, or better, it may be kept in solution together with ammonium nitrate. A solution of 34 % calcium nitrate and 32 % ammonium nitrate with a nitrogen content of 1 % and a crystallisation point of 4°C should be well fitted for distribution.

The mother liquor from the separation of calcium nitrate is neutralized in two or more steps with ammonia. During this neutralization calcium phosphate, calcium fluoride and different impurities are precipitated, and dissolved ammonium nitrate and ammonium phosphate are formed. This NP-slurry is concentrated by evaporation, granulated - if wanted with addition of other plant nutrients -, dried and screened. The undersize particles below 1.0-1.5 mm and oversize particles above 3,5 - 4.5 mm are recycled, the coarse fraction via a mill. The product is conditioned with kieselguhr or other agents for prevention of caking.

If we suppose that all nitrogen is originally present as liquid ammonia, the demand for cooling and steam will be more than covered by evaporation and oxidation of the NH_3. The demand for fuel oil is about 10 kg, and for electricity 70 kWH pr ton NP-product.

For a factory with a capacity of 85 000 tonnes NP-fertilizer 20 - 9 (20 % N, 9 % P) or 130 000 tonnes NPK 13 - 6 - 17, the investment is about 2.5 - 3.0 million $ and a corresponding nitric acid plant will amount to 1.5 - 2.0 million $. The necessary staff in a factory of this size will be 45 - 50 men.

The chemical composition of the product can be varied within wide limits and meet practically any agricultural requirement. The amount of water soluble phosphorous is normally about 30 % of the total phosphorous, and the citrate soluble amount 95 - 100 %. By further cooling the water soluble part can be increased to about 90 %.

Today the production and consumption of fertilizer made by the Odda process is concentrated in the northern and the middle part of Europe.

As a whole the fertilizing effiency corresponds to the same amount of plant nutrients given as straight materials. A somewhat slower action of the phosphorous may be found than compared with superphosphate.

In addition to being economic in production and covering all main agricultural demands, a concentrated, complete and ready-to-use fertilizer of the Odda process type will save money and labour.

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Agenda Item: D.4.2

Brazil

ESTABLISHMENT AND OPERATION OF A TROPICAL CENTER FOR THE STUDY OF FOOD TECHNOLOGY

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Stablishment and operation of a "Tropical Centre for the Study of Food Technology in the Instituto Agronômico, Campinas.

By

André Tosello

Diretor of Soil, Farm Mechanic and Technology Division of the Instituto Agronômico-Campinas.

1.- Introduction

Brazil with its populatior 75 millions peuple must be supplied with 150 thouzands tons of food daily, both of animal and vegetable origin. In one year the total need amounts to 50 millions tons. Such an amount of food is produced in different regions of Country, for that rea - son it must be stored and preserved to distribuite it normaly true the year and some times more than one year. Furthermore, it must be conditioned for transportation from the places where they are produced to the places where they are consummed, involving oftenly large distances. In Brazil this aspect of transportation is specialy impor - tant if one considers that those distances are not only large but communications are partly defficient.

It is obvious that nowadays, as in the past, on could not supplie the population with fresh foods only; be it of animal or vegetable nature. One the contrary the food must be processed, involving since simple treatments to keep the food from deterioration to more complicated procedures, to turn the raw materials in condition to be absorved by the human organism.

In a few years the population of Brazil will increased to hundred millions; the urban population will be much bigger and the production aereas will be farther out. The farmer will have to produce enough food to supply an increased number of peuples. The economical implications will be in sense that the agricultural regions will develope into specialised activities. The increased volume of production will be attained in more proper season.

It means that the problems of food supply will be more serious and to solve them will be required more technical knowledges, specialy in the field of food technology.

2.- Importance of the food Industry in Brazil

Brazil's food industry has developed vigorously thro ugh private enterprise and has attained great economic importance. There are 9,943 registered food factories. The value of manufactured food production in recent years has amounted to 25% of that of Brazil's total industrial production; a highly significant fact in view of the coun try's advanced stage of industrialization.

This industry has exceptional prospects for future development because of the country's virtually inexhaustible sources of raw materials of both vegetable and ani mal origin; the need standardize and cheapen the food - supplies of populations far agricultural and livestock production centres; because the food-manufacturing plants are permanent markets for the growing agricultural output; because these could utilize the area's surplus production of some agricultural commodities which are now wasted, since they are perishable foods that are sold fresh during the harvesting season at extremely low prices; because low-cost manufactured products would, in short, make it possible to raise the nutritional level of the population, which woul necessarily have a favourable impact on national economic development.

It is therefore the Government's duty, at both Fede - ral and State level, to promote the rational development of the food inusties by planning their economic develop - ment, providing technical guidance and settling their pro blems which could not be properly solved at the level of private enterprise.

3.- Factors hindering their development

Notwithstanding the strong impetus of Brazil's food industries, there are innumerable factors hindering their full rational development, inter alia: the complete lack of comprehensive theoretical and practical courses, at both mediun and higher levels, in order to train efficient technicians to work in the food industries. This has compelled the food industries to bring technicians from other countries at huge expense; to provide rudimentary empirical training, or to send people for training abroad.

The food industries lack the necessary laboratory e- quipment and qualified researchers to solve their technical problems that require scientific research .

It is not easy for these industries to obtain technical advice concerning the purchase or new equipment, expen

sion of that already existing in their factories, processing of new products, utilization of by products, improvement of their manufactured products, rational reduction of production costs, etc., owing to the scarcity of eccessible sources of technological information, namely, specialized technicians, technical libraries or experience gained by other countries.

The commercial rivalry existing among food manufacturers, who jealously guard as "industrial secrets" the experience they have acquired and the improvements introduced in the manufacture of their products obstructs technical development and creates methods of commercial competition which are detrimental to the quality of the products.

The lack of proper technical guidance impels manufacturers to use poor-quality raw materials so as to lower production costs, which obviouslaalso has a detrimental effect on the quality of manufactured products. In Brazil this occurs in the case of milk, oranges and tomatoes.

There is an urgent need for food inspectors,not only trained to apply punitive measures, but also with the necessary technical knowledge to advise industries as to how their technical and sanitary errors can be avoided.

4.- Efforts made by the government of the State of S.Paulo

In 1959, the State Department of Agriculture of S. Paulo, under the administration of Dr. José Bonifácio Coutinho Nogueira, being jully aware of the imperative necessity of promoting the introduction of up-to-date techni - que in the food manufacturing industries and of encoura - ging their development to a level compatible with their buge potentialities, designed the autor to stud the sta - blishment and operation of a Tropical Center for the Study of Food Technology.

The autor with the help of a group of local food technologists and Dr.Sherman Leonard, Food Technologist of University of California, specialy contracted for this work, drew up the plans and specifications for the various buildings laboratories and industrial pilot plants in accordance with Brazil needs and requirements. This is made,after careful study of the State of S.Paulo's availabilities of raw materials and potentialities as food manufacturing area, indicated the Instituto Agronômico of Campinas,of the State of S.Paulo Departament of Agriculture, as the most suitable cite for a Tropical Centre for the Study of Food Technology.

The State of S.Paulo has attained the highest level of agricultural development in Brazil.

5.- Activities program of the Tropical Centre.

The Tropical Centre of Food Technology is stablished for the purpose of conducting applied research, training professional experts and technicians to work efficiently in both private and governmental activities and imparting knowledge about Food Science and Technology.

The activities of the Tropical Centre will be of a national charater, without losing sight of the regional interests, and will be oriented towards: improving industrial systems of manufacture and preservation of foods vegetable and animal origin; rationalizing their storage and transport; avoiding wast or spoilage of foodstuffs owing to poor handling or overproduction; developing new types of cheap food products with a connected with food storage, processing, preservation and transport at the different economic and social levels, and the introduction of up-to-date techniques. Its ultimate goal is to contribute to Brazil's economic development.

In order to comply with these aims, the Tropical Centre of Food Technology will have, the following responsibilities;

1.- Coordination and promotion of scientific and technological research on the transport, storage, processing and preservation of foods of vegetable and animal origin.

2.- Scientific and technological research to help the domestic food industry solve its specific practical problems.

3.- Development of new low-cost food resources with a high nutritive value (especially,rich in proteins),to help solve the nutrition problem of the low-income population.

4.- Research on the effects of processing on the nutritive value of foodstuffs.

5.- Study of the traditional methods of food preservation (drying, salting and fermenting)so as to improve the techniques currently in use, and their extension.

6.- Development of pratical means of avoiding food wastage and spoilage.

7.- Maintenance of an information service for carrying out technological extension, publishing results obtained by research and providing technical information to food manufacturing industries.

8.- Collaboration with University and Medium Level Education for the training of Food Technologists with the necessary scientific and technical background to work efficiently in the food industries and government industrial, inspection and control services.

9.- Holding of short practical courses to include elementary in the food manufacturing industries who have had only empiric training.

10.- Promotions, coordination and improvement of efforts by government, universities and private enterprises in favour of the development and improvement of the country's food industries.

11.- Practical extension of industrial techniques , throug objetives demonstrations in the various pilot plants and laboratories.

12.- Bacteriological, chemical, phisical, acceptability, nutritive and other analyses, for the purpose of reconmending in confidence to each individual manufacturer, apropriate technological measures to improve his manufactured products and, at the some time, suggesting to government (either Federal or State) general technical standards for a better food control.

13.- Development of analytical methods that may be applied to the study of tropical foods.

14.- Study of the use food industry waste wherever this is economical feasible.

15.- Exchange of experience and results obtained from research, with different national or foreign organizations concerned with Food Technology.

6.- Instalations and laboratories

The government of São Paulo allocated the sum of aproximately US$2.000.000,00 out of its Regular Budget for constrution of Buildings, equipaments and scientific aparats. For operating of the Centre, The Special Funds

of the United Nations Organization will provide part of the expentidures.

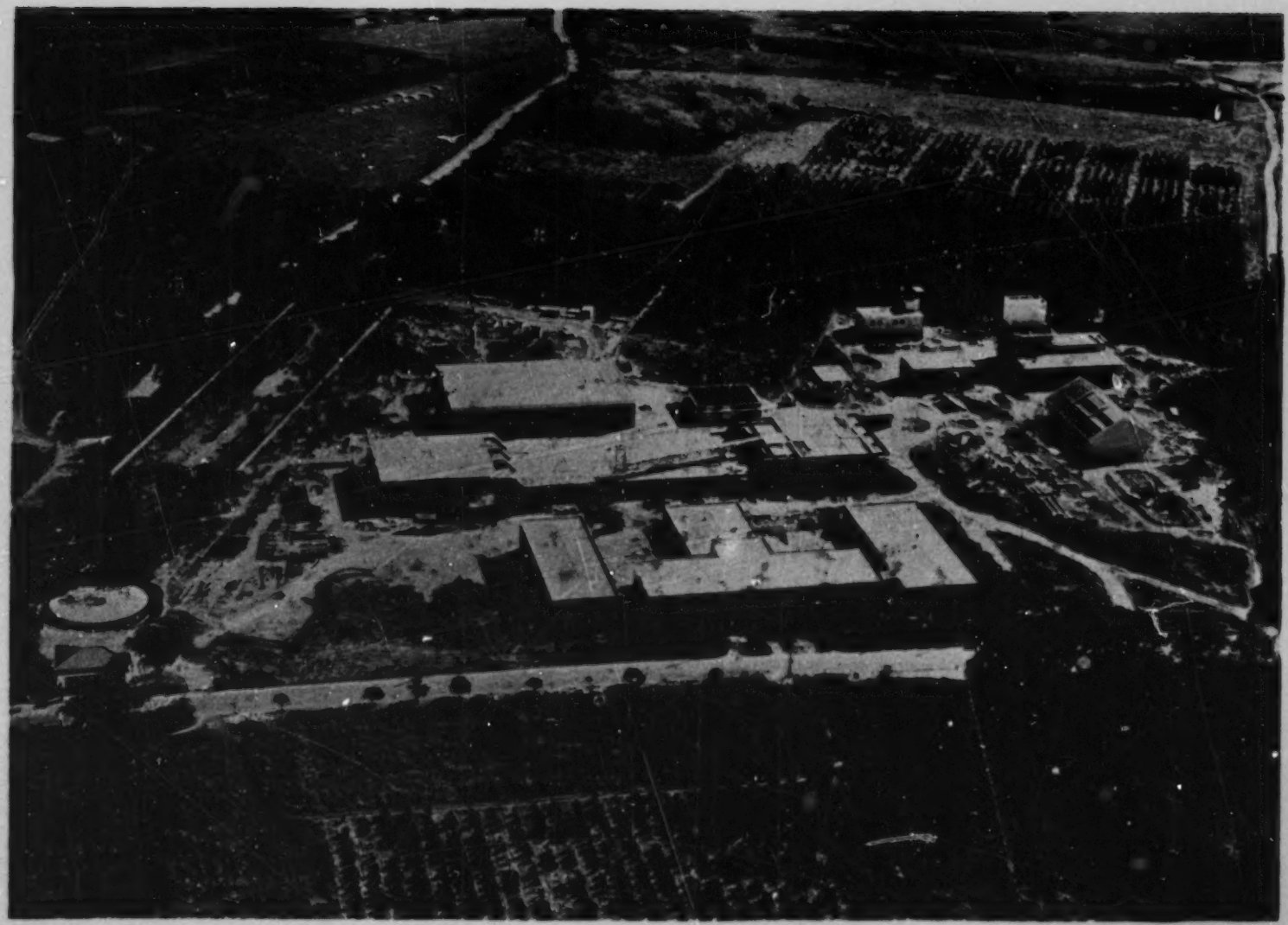
The Centre is located in a 65.000 square meter of land of the Instituto Agronômico - Estação Experimental "Theodureto de Camargo" - Campinas. Several Buildings are at present being ereted cover a total of 14.000 square meters, the constrution is almost completed.

This establisment, which will be the largest in South America devoted to research and technology connected with food of vegetable origin, will have the following pilot plants:- (1) canning, (2) Edible oils(Speller and solvent extraction), (3) Edible oil refining, (4) Fruit Juice Concentrates, (5) Refrigeration and Freezing, (6) Industrial Fermentation, (7) Grain ensilage, (8) Bakery, (9) Distillery of Alcohol used in Beverages.

It will also have following laboratories:-

Chemistry
Biochemistry
Microbiology
Engineering
Acceptability of Foodstuffs
Chromatography
Enzymology

The Food Technology Centre at Campinas will be an international Center for Tropical Food Technology research and also serve as a place for post graduate studies for technical men from other tropical countries.



Tropical Centre for the study of Food Technology in the Instituto Agronômico, Campinas

647 E - Oct. 1962 - 1,600

E/CONF.39/D/68
SUMMARY
9 Oct. 1962
ORIGINAL : ENGLISH

UNITED NATIONS CONFERENCE
ON THE APPLICATION OF SCIENCE AND TECHNOLOGY
FOR THE BENEFIT OF THE LESS DEVELOPED AREAS

SUMMARY

Agenda Item: D.4.2

Brazil

ESTABLISHMENT AND OPERATION OF A TROPICAL CENTER FOR THE STUDY OF FOOD TECHNOLOGY

By André TOSELLO,
Director,
Soil, Farm Mechanic and Technology Division,
Instituto Agronômico de Campinas,
São Paulo - Brazil

Papers have been contributed to the United Nations Conference on the Application of Science and Technology for the Benefit of the Less Developed Areas as working papers for that Conference. They are published as presented by the contributors, and the contents and the views expressed are those of the contributors. (See notes overleaf.)

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Sub-item D.4.2
Serial number 428

Establishment and operation of a tropical center for the study of food technology

by

André Toselb - Director of Soil, Farm Mechanic and Technology Division

Instituto Agronômico de Campinas - São Paulo - Brazil

SUMMARY

On of the basic problems in tropical countries is the production of enough food for their population. Most of these countries have an economy based on Agriculture and paradoxically, it is in this field that most of their failures become more apparent, being probably one of the basic causes of under development in such areas.

Food technology research is of exceptional importance for tropical countries. Numerous are the problems facing the investigator in this field. A short list of them would include: the study of preservation, storage, and handling of agricultural food products; the discovery of substitutes for wheat; research on edible oils, canned and frozen food, fruit juices and other products. These are practically unexplored areas in tropical food technology research.

Under the initiative of the Government of the State of São Paulo, a large center for food technology research is being created at the Instituto Agronômico, Campinas. The initial cost of this project is being estimated at about U.S.$3,000.000. This center will consist of a series of 15 buildings with very modern installations. Pilot plants will be available for the production of canned and frozen foods and juices; for dehydrated foods; for oil processing; for the production of distilled or fermented alcoholic drinks. Equipment will also be available for the investigation of methods of preservation, storage, and handling of cereals. Power, gas, water, and other facilities to be available at the Food Technology Center in Campinas will be equivalent to those needed for a city of about 5,000 in habitants.

The Food Technology Center at Campinas will be an international center for tropical food technology research and also serve as a place for post graduate studies for technical men from other tropical countries. Part of the expenditures for operating the center at Campinas will be met with funds granted by the United Nations Organization.

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Agenda Item: D.6.1

United Kingdom

THE HARVESTING, GINNING AND CLEANING OF COTTON

By Professor J.J. VINCENT,
M.Sc., M.Sc.Tech., F.T.I.

Papers have been contributed to the United Nations Conference on the Application of Science and Technology for the Benefit of the Less Developed Areas as working papers for that Conference. They are published as presented by the contributors, and the contents and the views expressed are those of the contributors. (See notes overleaf.)

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No. 1620
D.6.1.

The Harvesting, Ginning and Cleaning of Cotton

By Professor J. J. Vincent, M.Sc.,M.Sc.Tech.,F.T.I.

1. The most significant development in recent years in the harvesting of seed cotton i.e. the spinnable fibre (called lint) with the seeds still attached to it, has been the successful introduction in the United States of mechanical methods. Thus, in 1949 only 6% of the crop was mechanically harvested, with 9,500 machines in use, whereas in 1955 23% was harvested mechanically using 41,800 machines(1), while in 1961 this proportion had risen to 59%(2).

2. Harvesting machines fall into two classes: strippers and spindle pickers, the latter often being referred to merely as pickers. The first type of machine removes the bolls, as the seed-pods are called, by, in effect, combing them from the stems of the plant. The method does not discriminate between ripe, opened bolls and unripe ones and if any leaves are present on the plant at the time a large proportion of these will also be collected. Spindle pickers are more sophisticated machines and operate by bringing rotating spindles into contact with the lint itself which becomes exposed when the bolls ripen and open. The spindles are grooved, knurled or moistened so that the lint adheres to and becomes wrapped round them. The spindles travel on an endless belt and when loaded move to another part of the machine for the lint to be removed and stored. Both types of machine have been fully described by Smith(3).

(1) Brown, H.B. and Ware, J.O., Cotton, McGraw-Hill Book Company, Inc., 343,New York (1958).

(2) United States Department of Agriculture; Charges for Ginning Cotton etc., Season 1961-62 (May 1962).

(3) Smith, H.P., Farm Machinery and Equipment, McGraw-Hill Book Company, Inc., New York (1955).

3. The difference between the two types of machine in their ability to discriminate between seed cotton and foreign matter (called trash) is clearly shown in Table 1, where a comparison can also be made with the two methods of hand-harvesting employed - picking, where the aim is to select only ripe seed cotton, and snapping where the whole boll is removed by breaking it off at the stem. It will be seen that machine and hand picking produce substantially the same results but that machine stripping is distinctly less discriminating than snapping, both of the last two methods being much worse than the first two.

4. The proportion of cotton harvested by the various methods varies widely from region to region in the United States, being influenced by the particular system of land tenure, the nature of the farmland, the climate, the supply of seasonal labour, the level of wages, the price of cotton and other factors. Thus, in the 1957-58 season all the cotton was hand-picked in Virginia compared with only 1% in Oklahoma, where 50% was hand-snapped and 49% was machine-stripped, while in California 70% was machine-picked compared with virtually none in Alabama, Georgia, North and South Carolina, Tennessee and Texas(4).

5. In favourable conditions a mechanical picker, which may cost from 4,000 U.S. dollars for the simplest type without a tractor, up to 19,000 dollars for a two-row self-propelled one, will harvest the equivalent of a 500-lb. bale of cotton in about an hour; a mechanical stripper, which costs less, will gather a bale in half that time; the average hand-picker will take nearly 100 hours. These facts provide, however, only part of the information necessary to decide whether the use of mechanical harvesting will prove economic. If no change is made in the procedure at the next process, where the spinnable cotton fibre is removed from the seed, a process known as ginning, then, compared with hand-picked cotton, the machine-harvested cotton will contain more trash after ginning, be graded lower, and will command a lower price. Furthermore, an allowance must be made for the cotton that the machines fail to pick, which may be in the region of 10%.

6. Studies of this kind have been made by Crowe(5), by Butler and Streetman(6) and by Hedges and Bailey(7). They show how the break-even point depends

(4) United States Department of Agriculture; Charges for Ginning Cotton etc., Season 1957-58 (May 1958).

(5) Crowe, G.B., Mechanical Cotton Picker Operation in Yazoo-Mississippi Delta, Miss. Agr. Expt. Sta. Bul. 465 (1949).

(6) Butler, C.P. and Streetman, H.L., Economics of Mechanical Cotton Picking in South Carolina, S.C. Agr. Expt. Sta. Bul. 399 (1952).

(7) Hedges, T.R. and Bailey, W. R. Economics of Mechanical Cotton Harvesting, Calif. Agr. Expt. Sta. Bul. 743 (1954).

on the wage level for hand-picking, the intensity of use of the machine, and the yield per acre of the crop. Since these studies have been made the issue has been further complicated by the installation of lint-cleaning equipment at the ginneries, the number of such installations in the United States having increased from under 30 in 1948 to over 5,000 by 1961. According to Nickerson(8) the ginneries are much more efficient in removing leaf today than they were before the days of mechanical harvesting.

7. The defoliation of the cotton plant by chemical means before mechanical harvesting takes place is virtually essential, except where the onset of frost has already caused the leaves to fall, and it is also of assistance when hand-picking in making the bolls more accessible and in preventing their rotting. Successful defoliation is not easy to achieve since an insufficient quantity of defoliant will not cause the leaves to fall while an excess will kill the leaves prematurely before the natural processes causing them to fall have had time to be effective and the dead leaves will remain firmly attached to the plant. Moreover, the desirable quantity of defoliant to apply varies with the climate, the soil and the condition of the plant. A further aim is to prevent the subsequent growth of new foliage.

8. The research in this field has been described by Eaton(9). Among the most useful chemicals are calcium and monosodium cyanamides, which may be applied as dusts, and a wider range which may be used in the form of sprays - endothal, monosodium cyanamide, pentachlorophenol, potassium cyanate, magnesium chlorate hexahydrate, sodium chlorate, sodium chlorate-pentaborate, sodium monochloro-acetate. The addition of amino triazole to some of these has helped to prevent the re-growth of foliage.

9. Mechanical harvesting has not so far made any headway outside the United States, though a few machines have been exported to Russia, because, on the whole, a good supply of seasonal labour has been available for hand-harvesting in the other cotton-growing countries. The opportunity that hand-picking offers of producing cotton relatively free from foreign matter is not, however, always seized. As regards avoiding the obvious contaminants such as leaf, stalk and sand, the correct procedure is largely a matter of good organisation, but an additional requirement is the appreciation of how

(8) Nickerson, Dorothy. Recent Trends in Trash Content of Raw Cotton Create a Problem in Grade Standards. Cotton Research Clinic, National Cotton Council of America, 8, (1962).

(9) Eaton, F. M., Physiology of the Cotton Plant, Ann. Rev. Plant. Physiol., 6, pp.299-328 (1955).

much harm can be done in processing and in the final product (cloth or garment) by the presence of minute amounts of any foreign fibres such as jute, which may originate from the sacks in which the seed cotton is placed, or specks of tar which may arise from the use of bituminised paper sacks for the same purpose.

Ginning

10. The machines employed for the separation of the lint from the seed are called gins and are of two kinds: saw-gins and roller-gins. In the first the lint is torn from the seed by the teeth of rapidly revolving circular saws; in the second the lint makes frictional contact with a rotating roller, covered with leather or similar material, and is pulled from the seed while the latter is held back by a knife-edge. The saw-gin is much more productive than the roller-gin but may damage finer and longer lint and it is for such cottons that the McCarthy type of roller-gin is mainly used. Roller-gins can also be employed for shorter lint attached to smooth seeds and are widely used in India for this purpose, but as many are of a relatively primitive type and liable to crush the seed, producing stained cotton, their use is undesirable.

11. The advent of mechanical harvesting in the United States has greatly stimulated the elaboration of the saw-gin, to such an extent that the actual removal of the lint from the seed is only one of six operations that take place when the seed cotton is processed in the most modern machines(10). The need for improvement arose from two features of mechanical harvesting: the application of moisture to facilitate spindle-picking and the presence with both types of mechanical harvesting of abnormal amounts of trash. If the seed cotton is too damp, ginning will be unsatisfactory because of clogging of the saws, cutting of the lint or its compacting into small lumps. The modern gin is therefore equipped with arrangements both for drying and cleaning. These facilities are also obviously of value for hand-picked cotton that is damp or contains excessive trash, but arrangements can be made to by-pass them when ginning clean, dry hand-picked cotton so as to avoid unnecessary processing.

12. The full sequence of operations is as follows. The seed cotton to supply a battery of 4 gins is sucked pneumatically from the delivery or storage containers and fed at a controlled rate to the first hot-air dryer. The partial drying at this stage facilitates the cleaning that takes place before lint separation. Larger pieces of foreign matter are removed by revolving toothed cylinders and smaller pieces by threshing over screens. After further drying the cleaning is repeated before the material is distributed to the individual gins where further drying and cleaning take place before the lint is separated from the seed. Finally, the lint itself is cleaned after separation from the seed and the cleaned lint is conveyed pneumatically to the presser and baler.

(10) Brown, H.B. and Ware, J. O., Cotton, McGraw-Hill Book Company, Inc., 374, New York (1958).

13. The whole process is highly automatic; the covering, tying and removal of the bales are almost the only operations to be performed by hand. Large quantities of air are used for conveying, about 60% of the total power for ginning being taken by the fans.

14. The foregoing account provides a strong contrast with the description given by Tippett(11) of the ginneries of the Sudan Gezira Board where all the cotton is processed on roller-gins and the feeding of seed cotton to the gins, the collection of lint, its feeding to the bale press and its trampling, are all done by hand or foot. In the report cited Tippett makes ten recommendations(12) concerning the initiation of research into ginning in the three countries concerned. He places particular emphasis on the need for a systematic study of the effects of different gin settings and conditions, with particular reference to roller-gins.

15. Much research on saw-gins has already been done at the U.S. Department of Agriculture Cotton Ginning Laboratory at Stoneville, Miss., and more recently at additional laboratories at Mesilla Park, N. Mex., Chickasha, Okla., and Clemson, S.C. Among the questions investigated has been the influence of seed-roll density and saw speed on the quality of the lint and on the ginning capacity and power requirements. The problem of handling trashier cotton is becoming more acute and continuing research is necessary into how to improve the cleaning of cotton during ginning without impairing its value for later processing.

Cleaning

16. Most of the cleaning of the lint - in the absence of lint-cleaners at ginneries, all the cleaning - is performed at the spinning mill in the course of the opening process, a necessary preliminary to yarn manufacture, in which the compressed cotton from the bale is loosened, opened up, blended and made into a relatively uniform fleece or lap. Even where lint-cleaners are now widely used at ginneries, as in the United States, an appreciable amount of trash remains to be removed by the spinner. Nickerson(13) estimates that the average non-lint content of the U.S. 1961 cotton crop lies between 1½% and 10%, depending on the grade.

17. Considerable progress has been made in recent years and there are now on the market several makes of opener with greatly improved cleaning powers. It is claimed for one that it is equivalent to four beaters of the traditional type. Another important factor is that the amount of good lint rejected with the trash should be kept to a minimum. In this respect, too, these modern

(11) Tippett, L.H.C., Report on a Tour of Cotton Ginning Industries in the Sudan, Uganda and Tanganyika. Empire Cotton Growing Review, 197,(1960).

(12) Tippett, L.H.C., ibid, 206.

(13) Nickerson, Dorothy. Recent Trends in Trash Content of Raw Cotton Create a Problem in Grade Standards. Cotton Res. Clinic, National Cotton Council of America, 8, (1962).

machines show to advantage. Tests on another make of machine showed that there was only 17% and 13% of lint in the waste rejected at the two cleaning points compared with 33% in the waste from a conventional opener.

18. These improvements have come about as the result of researches, carried out at the Shirley Institute(14),(15) and elsewhere(16), into the effectiveness of conventional opening machines and into the differences in aerodynamic properties between tufts of cotton of various sizes and the various types of trash. It was shown that the common practice of the spinner who cleaned his cotton more thoroughly by merely passing it through an additional machine of the same general type was very inefficient. Instead, the cotton should be opened up progressively into smaller and smaller tufts if any marked improvement were to be made. When this was done it was possible to make use of the differences in aerodynamic properties to cause, with the aid of carefully controlled air currents, the lint and trash to take divergent paths through the opening machine, so achieving the desired separation.

19. One danger with these newer opening machines is that in opening up the cotton to the extent required damage may be done to the fibres, particularly those of long staple, and any potential user should check that they are suitable in this respect for the cottons he wishes to process.

20. Further cleaning takes place in carding, a process in which the lap produced by the last opening machine is teased out, almost fibre by fibre, into a thin film, or web, before being condensed into a soft continuous rope called card sliver. During carding, many small particles of trash are eliminated which have been embedded in tufts of cotton and have hitherto failed to be removed. Nevertheless, sufficient of the lighter particles, consisting of fragments of leaf, stalk or seed-coat, may still be present to cause difficulty or extra expense in later processes.

21. This residual waste is now being dealt with effectively by fitting carding machines with web purifiers(17). Each unit consists of a pair of precision-ground hardened cylindrical pressure rollers between which the card web passes before being condensed into a sliver. The pressure between the rollers is such as to crush the trash particles without damaging the cotton fibres. Much of the pulverised trash falls out before the cotton is spun into yarn and any remaining is completely eliminated in finishing.

(14) Peirce, F.T., Kelly, J.F., and Coleman, Mary H., A Survey of Blowroom Practice, Journ.Text.Inst., 46, T 78 (1955).

(15) Bostock, W., Freeman, S.M., Shorter, S.A., and Williams, T.C., An Aerodynamical Study of the Opening and Cleaning of Cotton by Existing Machinery, Journ.Text.Inst., 46, T 171 (1955).

(16) Higuchi, K., An Experimental Study of Blowroom Machinery, Journ.Soc. Text. and Cell. Ind. Japan, 13, 783, 877, (1957); 14 26, 30, 467, 551, 627, 693(1958); An Aerodynamic Study of Cotton Tufts in Blowroom Machinery, J.Text.Mach.Soc.Japan, 11, No.7,9; No.8,14,No.11,11.(1958).

(17) Clinch, H.H. The Crosrol Cotton Web Purifier, Cotton Research Clinic, National Cotton Council of America, 61, (1962).

22. The rollers are arranged so that a uniform pressure is achieved across the whole width and knife-edged scraper blades keep them clean. Their surface speed is the same as that at which the web leaves the card but it is less than that of the rollers which draw away the condensed web. In this way the fibres are disturbed and the impurities loosened.

Conclusion

23. The implications of the foregoing for any particular country will depend upon its general state of development, whether it is a cotton-growing or a cotton-using country or both, its supply of capital and labour and their relative costs, and the state of development of its light engineering industry. For this reason local studies will be necessary to determine how far the developments outlined above are appropriate to the particular circumstances prevailing at the time, and as these may change rapidly continuing assessment will be necessary.

24. The experience of the United States should be freely made use of and it should be possible to draw valuable conclusions, concerning harvesting in particular, from the diversity of practice between the different regions noted in para. 4, applicable to countries covering a wide range of stages of development.

25. Where shortage of capital or foreign currency prevents the purchase of machinery from abroad local manufacture may be possible in certain instances. All capital expenditure should be carefully examined from the standpoint of avoiding the purchase of obsolescent machinery.

26. The responsibility for producing clean yarn is shared between the grower, the ginner and spinner. The most economical way of apportioning that responsibility among them is an important but complex problem the solution to which will vary from country to country.

27. Since cotton is the most widely used of industrial fibres there will be few countries to whom the developments outlined in this paper are of no concern. Appreciable research effort in each country would appear to be justified and a sharing of experience desirable.

ANNEX

Table 1.

Average weight of harvested material (seed cotton plus trash) necessary to produce a 500-pound bale of cotton as supplied to the spinner; United States, season 1957-8[a].

Harvesting method	Weight of seed cotton plus trash (lb)
Hand-picked	1,430
Hand-snapped	1,989
Machine-picked	1,492
Machine-stripped	2,261

(a) United States Department of Agriculture; Charges for Ginning Cotton etc., Season 1957-58 (May 1958)

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ON THE APPLICATION OF SCIENCE AND TECHNOLOGY
FOR THE BENEFIT OF THE LESS DEVELOPED AREAS

SUMMARY

Agenda Item : D.6.1

United Kingdom

THE HARVESTING, GINNING AND CLEANING OF COTTON

By Professor J.J. VINCENT,
M.Sc., M.Sc. Tech., F.T.I.

Papers have been contributed to the United Nations Conference on the Application of Science and Technology for the Benefit of the Less Developed Areas as working papers for that Conference. They are published as presented by the contributors, and the contents and the views expressed are those of the contributors. (See notes overleaf.)

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The Harvesting, Ginning and Cleaning of Cotton

By Professor J. J. Vincent, M.Sc.,M.Sc.Tech.,F.T.I.

SUMMARY

The most significant development in recent years in the harvesting of cotton has been the successful, but far from universal, application of mechanical methods in the United States. Two classes of machines are employed: spindle-pickers and strippers. The first are more discriminating in their action than the second but the second are more appropriate for use in certain circumstances. In favourable conditions machine picking produces substantially the same results as hand picking, so far as yield of lint per pound of seed cotton is concerned, but the machine may leave some cotton unpicked. Labour productivity may be increased 200-fold by the use of strippers and 100-fold by spindle-pickers.

The economics of mechanical harvesting is very complex, but several studies made in the United States are available as guides. The relative importance of the various factors is continually changing and the situation is further complicated by the installation of lint-cleaners at the ginneries.

In general, mechanical harvesting demands prior defoliation of the cotton plants, which is only accomplished successfully by the application of certain chemicals, such as calcium cyanamide, in carefully controlled quantities.

The advent of mechanical harvesting has greatly stimulated the elaboration of the saw-gin to enable the seed cotton to be dried and cleaned before the lint is separated from the seed and to enable the lint to be cleaned afterwards. The whole process has also been made highly automatic.

Considerable improvement has been made in recent years in the machines used by spinners for the preliminary opening and cleaning of cotton. These improvements are a consequence of the researches done at the Shirley Institute and elsewhere into the effectiveness of conventional opening machines and into the difference in aerodynamic properties between tufts of cotton of various sizes and the various types of trash. One of the new makes of opener does the work of four conventional machines and another make rejects only about half as much good lint along with the extracted waste compared with a conventional machine.

Certain types of residual waste, still present aftecarding, may be pulverised by passing the card web between a pair of precision-ground hardened cylindrical pressure rollers. The crushed waste falls away from the cotton before it is spun into yarn.

The implications of the foregoing for any particular country depend upon its general state of development, whether it is a cotton-growing or a cotton-using country or both, its supply of capital and labour and their relative costs, and the state of development of its light engineering industry. Local and continuing studies will be necessary to determine how far the developments outlined above are appropriate at any particular time. The experience of the United States should be freely drawn on, particular attention being paid to the diversity of practice in different regions of that country.

Since cotton is the most widely used industrial fibre many countries will be concerned with the developments outlined above. Appreciable research effort in each country would appear to be justified; a pooling of results would be desirable.

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CONFÉRENCE DES NATIONS UNIES
SUR L'APPLICATION DE LA SCIENCE ET DE LA TECHNIQUE
DANS L'INTÉRÊT DES RÉGIONS PEU DÉVELOPPÉES

Point de l'ordre du jour : D.4.2 France

ETUDE SUR LA PERMEABILITE A LA VAPEUR D'EAU DES MATIERES PLASTIQUES EN FEUILLES SOUPLES ET MINCES, EN FONCTION DE L'EPAISSEUR, DE LA TEMPERATURE, DE L'HUMIDITE

Par Ph. BLANCHE,
Ingénieur au Laboratoire général
pour emballages

Les mémoires rédigés à l'intention de la Conférence des Nations Unies sur l'application de la science et de la technique dans l'intérêt des régions peu développées sont des documents de travail. Ils sont publiés tels qu'ils ont été présentés et les assertions et opinions qu'ils contiennent sont celles de leurs auteurs.

NOTES

1. Les mémoires choisis pour être présentés en séance générale pourront être obtenus à la Conférence dans les langues officielles suivantes des Nations Unies: anglais, espagnol, français et russe.

2. Les mémoires choisis pour être présentés en séance spéciale pourront être obtenus à la Conférence dans l'une seulement de ces langues officielles.

3. Les résumés qui ont été rédigés pour tous les mémoires par les auteurs de ceux-ci pourront être obtenus à la Conférence dans les langues officielles suivantes des Nations Unies: anglais, espagnol, français et russe.

4. Préalablement à la Conférence, les mémoires et les résumés ne feront pas l'objet d'une distribution générale mais d'un tirage restreint destiné aux délégations des gouvernements et autres participants officiels. La publication et la distribution générale des actes de la Conférence s'effectueront conformément aux règles et pratiques agréées par le Comité des publications des Nations Unies.

ETUDE SUR LA PERMEABILITE A LA VAPEUR D'EAU DES MATIERES PLASTIQUES EN FEUILLES SOUPLES ET MINCES, EN FONCTION DE L'EPAISSEUR, DE LA TEMPERATURE, DE L'HUMIDITE

par Ph. BLANCHE, ingénieur au Laboratoire général pour Emballages

- P R E L I M I N A I R E S -

L'utilisation de plus en plus importante des matières plastiques comme matériau de protection contre l'humidité implique la connaissance, aussi précise que possible, de la perméabilité à la vapeur d'eau de ces matériaux dans diverses conditions.

Cette connaissance du taux de perméabilité est essentielle pour déterminer le temps d'efficacité de la protection, la quantité de deshydratant à placer dans le volume à protéger.

Elle doit aussi permettre à l'utilisateur de fixer son choix sur la nature du matériau à utiliser et sur l'épaisseur à adopter.

Ces considérations nous ont amené à entreprendre une étude dont le but est de déterminer le taux de perméabilité à la vapeur d'eau des matières plastiques en film les plus utilisées, en fonction de leur épaisseur, de la température et de l'hygrométrie.

I - CHOIX D'UNE METHODE

Avant de commencer les mesures, il a été nécessaire de faire le point sur les différentes méthodes utilisées. La documentation et les contacts avec différents Laboratoires ont fait ressortir une seule et même méthode générale qui consiste à séparer deux milieux, l'un sec et l'autre humide, par le film dont on cherche à déterminer la perméabilité à la vapeur d'eau.

Mais il existe de nombreuses variantes à cette méthode générale. Ces variantes portent d'une part, sur la fixation du film sur le porte-éprouvette, d'autre part, sur les moyens de créer les milieux sec et humide.

Il a donc été nécessaire d'expérimenter toutes ces diverses variantes afin de fixer notre choix ; tel a été le but de la première étude.

1) Méthode de la cuvette sèche

a) à joint de cire

Sur une cuvette en aluminium contenant du Ca Cl2 anhydre l'on place l'échantillon circulaire à essayer. L'on assure sa fixation et l'étanchéité en coulant un joint de cire sur l'échantillon le long du bord de la cuvette. Après avoir pesé cet appareil à 0,001 g près, on le place dans une étuve humide. Toutes les n heures, l'on repése l'appareil jusqu'à ce que l'accroissement de poids entre deux pesées soit constant.

Ce procédé a des inconvénients :

- la confection du joint de cire demande une certaine dextérité,
- il est difficilement utilisable pour les matériaux revêtus d'un corps dont le point de fusion est inférieur au point de fusion de la cire,
- le joint de cire a parfois tendance à se contracter, à se décoller des bords de la cuvette et l'étanchéité n'est plus assurée,
- enfin, certaines cires peuvent être légèrement hygroscopiques.

Le premier inconvénient : dextérité : est éliminé par l'entraînement de l'opérateur.

Le deuxième inconvénient : incompatibilité avec certains matériaux enduits : disparaît du fait que nous n'étudierons que les "matières plastiques" au sens actuel et restreint du terme.

Le troisème inconvénient : décollement du joint : a été éliminé par l'adoption d'une cuvette à gorge dont le profil a été soigneusement déterminé.

Le quatrième inconvénient: cire hygroscopique : a été écarté par l'adoption, après de nombreux essais, d'un type de cire minérale particulier : MOBILWAX n° 5.

b) à anneau vissé

Une solution consiste à remplacer le joint de cire par un anneau à filetage interne que l'on visse, le bord de la cuvette comportant un filetage externe.

De nombreux essais ont fait ressortir les inconvénients suivants :

1/ froissement et détérioration du film au moment du serrage de l'anneau,

2/ après un séjour de quelques heures en étuve, l'anneau s'est dilaté et légèrement desserré,

3/ impossibilité à peu près totale d'obtenir une étanchéité parfaite du filetage,

4/ grippage du filetage extrêmement fréquent,

5/ conséquence des inconvénients 2 et 3 : grands dispersion des résultats.

A côté de tous ces inconvénients, apparaît un avantage : mise en oeuvre beaucoup plus facile que la confection du joint de cire, mais cet avantage ne peut évidemment compenser les inconvénients 2 et 3.

L'on a essayé de supprimer les inconvénients 2 et 3 en plaçant, dessus et dessous l'échantillon, divers joints :

- joints en élastomère de différents types, de différentes duretés,
- joints métalliques
- joints en matière plastique

Aucun résultat satisfaisant n'a été obtenu.

c) Cuvette à anneau vissé contenant un vase à tare

L'on a essayé de ne pas mettre le Ca Cl2 directement en contact avec la cuvette, mais dans un petit récipient en verre placé dans la cuvette. Ce procédé aurait eu l'avantage de permettre de peser uniquement le récipient en verre. Mais, outre tous les inconvénients de la cuvette à anneau vissé, l'on ajoutait celui de devoir, à chaque pesée, démonter et remonter l'appareil.

2°) Méthode de la cuvette humide

L'on a essayé de remplacer le Ca Cl2 dans la cuvette par de l'eau, et de placer l'ensemble dans une atmosphère sèche. L'on ne mesurait plus alors un gain de poids, mais une perte de poids.

Les inconvénients se sont révélés nombreux.

Tout d'abord, il était nécessaire d'utiliser une cuvette à anneau vissé. Ensuite il a été évidemment impossible de faire varier les tensions de vapeur de chaque côté du film de façon précise. A 40 °C, par exemple, on a constaté que, puisqu'il y a saturation dans la cuvette, des gouttelettes d'eau se déposaient sur la face interne du film.

Si l'on opère au bain de sable ou au bain d'huile, il faut avant chaque pesée essuyer soigneusement la cuvette, première cause d'erreur, d'autre part, il est nécessaire que la tension de vapeur du milieu extérieur ambiant soit constante, pendant toute la durée de l'essai. Si la cuvette est à 40 °C et que dans le Laboratoire la température et l'hygrométrie passent de 15 °C et 80 % H.R. à 30 °C et 70 % H.R. dans la même journée, les tensions de vapeur passent de 10 mm Hg à 22 mm Hg pour le milieu extérieur, mais la tension de vapeur à l'intérieur de la cuvette est toujours de 53 mm Hg environ.

Aucune mesure précise ne peut être faite dans ces conditions.

Cet inconvénient ne peut d'ailleurs être éliminé en plaçant la cuvette dans le four "sec" à 40 °C. L'air contenu dans ce four n'est sec que par rapport à l'humidité qu'il contenait avant la mise en route du chauffage. Si cet air était à 20 °C et 65 % H.R. au moment de la mise sous tension du four, à 40 °C il sera encore à 20 % H.R. et ne pourra descendre plus bas que si les circonstances atmosphériques s'y prêtent. Mais, toujours pour les mêmes raisons, l'humidité relative ne pourra remonter dans ce même four, au cours de la même journée sans que l'on puisse intervenir.

3°) Méthode du sachet

L'on a enfin essayé de placer un sachet de Ca Cl2 dans une poche constituée par le film à essayer. Le tout étant ensuite placé dans une étuve humide. Ce procédé a l'avantage de permettre d'essayer des surfaces relativement importantes.

Mais les deux inconvénients principaux sont les suivants :

Le sachet ou la capsule perforée contenant du Ca Cl2 est en contact direct avec une portion de la surface du film, alors qu'il faudrait pouvoir placer l'agent deshydratant au centre du volume formé par la poche.

Ce procédé ne permet pas de tracer la courbe de perméabilité du film dans le temps puisque pour peser l'agent deshydratant, il faut détruire le film. La poche est, en effet, trop importante pour passer sur le plateau d'une balance analytique. Si l'on en réduit les dimensions on élimine, en même temps, l'avantage de la méthode.

A la suite des essais que nous avons effectués selon ces différents modes opératoires, nous avons décidé de retenir la méthode de la cuvette sèche, à gorge et à joint de cire.

II - DETERMINATION DE L'ENCEINTE D'ESSAI

A partir du moment où l'on a choisi d'utiliser la cuvette sèche à joint de cire, il devient évident que les meilleures enceintes d'essais possibles, devraient être constituées par des chambres à humidité et température variables et contrôlables. De telles chambres existent actuellement au L.G.E. Nous nous sommes donc attachés, en second lieu, à déterminer :

1°) le moyen de mesurer aussi exactement que possible les températures sèche et humide d'une enceinte;

2°) la gamme des H.R. et des températures qu'il est possible d'obtenir avec les étuves du L.G.E.

* * *

a) Mesure de l'humidité et de la température dans une enceinte de 250 dm3, ventilée

En ce qui concerne la température sèche, aucun problème ne se posait. La difficulté est apparue lorsqu'il s'est agit de mesurer précisément la température humide ou l'humidité.

L'hygromètre à cheveux a été, dès l'abord, écarté.

Des essais assez nombreux ont été faits à l'aide d'une cellule au chlorure de Lithium. Un tel appareil semblait facile à utiliser et est peu coûteux. Malheureusement, la cellule au chlorure de Lithium vieillit très vite, les étalonnages sur solutions de H_2S ou de sels saturés doivent être extrêmement fréquents ; et tous les auteurs ne sont pas d'accord sur les courbes d'humidité obtenues en fonction de la température et des sels utilisés.

C'est pourquoi nous n'avons pas retenu ce moyen de contrôle.

Après la cellule au chlorure de Lithium nous avons essayé un appareil de fabrication suisse, qui nous avait été prêté par le Comptoir Général des Gommes. Cet appareil, extrêmement coûteux, est basé sur les variations de résistivité d'un semi-conducteur en fonction de l'humidité.

Cet appareil nous a paru extrêmement simple d'emploi, sa mise en oeuvre est rapide, sa précision paraît suffisante. Malheureusement, nous n'avons pu l'avoir à notre disposition que durant une quinzaine de jours, ce qui nous a empêché d'observer sa constance dans le temps. Un tel appareil est actuellement en service à la Société Professionnelle des Papiers de Presse, et nous espérons avoir d'ici douze à dix-huit mois des renseignements sur sa constance de précision.

Les appareils à point de rosée ne nous ont pas paru d'une utilisation commode. Il ne nous restait plus que le psychromètre. Nous pensons obtenir par ce moyen une précision suffisante en utilisant des thermomètres de longueurs différentes selon les gammes de températures auxquelles nous travaillerons.

Les tables de Carrier, comme la formule de Sprundt, ont été établies pour des vitesses de veine d'air de 2 à 3 mètres par seconde. Il est certain que dans nos étuves de 250 dm3 nous n'avons pas réalisé ces conditions. Nous sommes en milieu essentiellement turbulent. Cependant, en dehors du psychromètre, nous ne voyons pas actuellement d'autres moyens suffisamment précis et suffisamment peu coûteux de mesurer les différences entre tension de vapeur saturante et tension de vapeur partielle.

C'est pourquoi nous avons décidé d'utiliser un psychromètre à poste fixe sur l'enceinte, avec enregistrement. Nous utiliserons des thermomètres au 1/10 dont les limites de graduations seront fonction des températures d'essai.

Enfin, le psychromètre à poste fixe sera périodiquement vérifié par un psychromètre-étalon à aspiration, établi d'après ASSMANN et décrit dans le projet de norme DIN 50 012.

b) Détermination des gammes H.R. et degrés C qu'il est possible d'obtenir dans les étuves 250 dm³ du L.C.E.

La température ambiante de l'atelier où sont placées ces étuves ne descendant jamais en dessous de 15 °C et montant à 30 °C l'été, la limite inférieure de température humide est très vite atteinte. Pratiquement, il est à peu près impossible d'afficher une température humide inférieure à 25 °C. Durant deux mois cette limite remonte même à 30 °C. C'est pourquoi avec le matériel dont nous disposons actuellement, il nous est impossible de travailler à des humidités inférieures à 50 % H.R. en dessous de 35 °C.

Nous avons longuement essayé divers moyens pour dessécher l'air des enceintes. En particulier, nous avons essayé d'amener une conduite d'air froid depuis le caisson 1 m3 froid jusqu'à l'étuve humide. Aucun résultat durable n'a été obtenu.

Nous avons donc décidé d'abandonner provisoirement l'étude des P.V.E. en fonction des variations de H.R. et de degrés C jusqu'à ce que nous disposions d'une étuve avec source froide.

Nous avons alors décidé d'adopter comme conditions d'essais : 38 °C et 93% H.R. ± 3 %. Cependant au cours du 2ème semestre 1962, le L.C.E. recevra un caisson de 1 m3 froid-chaud-humide dont les gammes de température et humidité permettront de réaliser d'autres conditions.

III - CHOIX DES MATERIAUX A ESSAYER

Après avoir déterminé :

1/ le mode opératoire
2/ l'enceinte à utiliser
3/ les appareils de mesure à utiliser
4/ la température et l'humidité relative auxquelles l'on commencerait les essais,

il restait à adopter une gamme de matériaux. Les matériaux les plus utilisés sont actuellement : le polyéthylène haute pression en épaisseur de 3 à 25/100, le chlorure de vinyle en épaisseur de 10 -15-30/100, le rilsan dans les mêmes épaisseurs que le polyéthylène.

IV - PREMIERES MESURES

Les premières mesures ont été effectuées au cours du mois de Juin 1962, à 38°C ET 93 %HR sur des polyéthylène haute pression, d'épaisseurs : 15/100 - 4/100.

Les quelques résultats obtenus sont consignés dans les deux tableaux ci-après.

Dans le mois de Septembre 1962, toute une série de mesures systématiques va être entreprise dont le dépouillement des résultats apparaîtra au début de 1963.

TABLEAU I - E = 15/100

	24 h	24 h	24 h	24 h	24 h	24 h	24 h	24 h	24 h	24 h	24 h	24h	24h	24h	MOYENNE
1	0,015	0,063			0,028	0,042	0,055								0,021
2	0,015	0,080			0,027	0,025	0,026								0,024
3	0,016	0,079			0,010	0,025	0,033								0,023
4	0,011	0,062			0,026	0,021	0,018								0,019
5	0,012	0,064			0,025	0,033	0,035								0,024
6	0,011	0,061			0,015	0,015	0,010	0,019		0,044					0,016
7	0,011	0,061			0,025	0,003	0,023	0,020		0,037					0,017
8	0,015	0,072			0,026	0,022	0,029	0,029		0,078					0,024
9	0,007	0,066			0,025	0,017	0,024	0,022		0,065					0,020
10	0,012	0,064			0,038	0,011	0,022	0,030		0,076					0,023
11	0,028	0,022	0,032	0,050											0,027
12	0,017	0,019	0,023	0,038											0,024
13	0,034	0,111													0,032
14	0,019	0,019	0,022	0,033											—
15	0,085	0,144													
16		0,060		0,056		0,041			0,050						0,020
17		0,061		0,052		0,037			0,044						0,019
18		0,059		0,056		0,046			0,047						0,020
19		0,057		0,067		0,025			0,046						0,019
20		0,065		0,048		0,037			0,058						0,020

TABLEAU II - E = 4/100

1	0,049	0,164	0,048	0,050	0,045	0,066	0,138						0,051
2	0,064	0,202	0,067	0,046	0,059	0,070	0,156						0,060
3	0,100	0,381	0,122	0,121	0,105	0,131	0,342						0,118
4	0,037	0,201	0,064	0,065	0,052	0,077	0,184						0,061
5	0,053	0,202	0,061	0,065	0,054	0,075	0,177						0,057
6	0,048	0,182	0,054	0,058	0,050	0,070	0,164						0,057
7	0,058	0,209	0,060	0,063	0,058	0,083	0,195						0,066
8	0,054	0,209	0,076	0,051	0,062	0,074	0,182						0,064
9	0,049	0,182	0,059	0,059	0,046	0,075	0,165						0,057
10	0,047	0,178	0,058	0,053	0,042	0,072	0,165						0,056
11		0,272		0,156		0,141	0,200						0,070
12		0,247		0,141		0,133	0,179						0,064
13		0,543		0,277		—	—						—
14		0,226		0,144		0,120	0,173						0,060
15		0,205		0,131		0,111	0,171						0,056
16 à 20		/		/		/	/			/			/

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France

THE PERMEABILITY TO WATER VAPOUR OF THIN, FLEXIBLE PLASTIC FILMS, AS A FUNCTION OF THICKNESS, TEMPERATURE AND HUMIDITY

By Ph. BLANCHE,
Engineer,
Laboratoire général pour emballages
(Packaging Laboratory)

Papers have been contributed to the United Nations Conference on the Application of Science and Technology for the Benefit of the Less Developed Areas as working papers for that Conference. They are published as presented by the contributors, and the contents and the views expressed are those of the contributors. (See notes overleaf.)

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THE PERMEABILITY TO WATER VAPOUR OF THIN, FLEXIBLE PLASTIC FILMS, AS A FUNCTION OF THICKNESS, TEMPERATURE AND HUMIDITY

By
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(PACKAGING LABORATORY)

The increasing use of plastic films in packaging as a protection against humidity, demands a fairly accurate knowledge of their permeability to water vapour under varying conditions. Knowledge of the permeability rate is essential to determine the period for which the film is an effective protection, as well as the calculated quantity of dehydrating agent to place in the volume to be protected. It should also enable the user to choose the thickness and type of plastic film which suits the function and its duration.

All these considerations led the Packaging Laboratory to undertake an investigation with the double purpose of determining:

(1) the simplest and surest method of determining the permeability to water vapour of film barrier materials

(2) the permeability rate of polythene films as a function of:

- (a) thickness of film
- (b) temperature
- (c) relative humidity, hence of the partial vapour pressure at a given temperature.

The first results are given in two tables, but tests are continuing. Polythene has been chosen for the laboratory tests simply because it was easily obtainable in a sufficiently wide range of thicknesses. The programme of the Packaging Laboratory, however, includes investigations into other barrier materials used for packaging.

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Agenda Item: D.8.1

South Africa

RECENT ADVANCES IN THE EXTRACTION OF URANIUM FROM SOUTH AFRICAN ORES

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RECENT ADVANCES IN THE EXTRACTION OF URANIUM FROM SOUTH AFRICAN ORES

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Introduction

1. The South African Uranium Industry was established with the utmost haste and urgency. At the time when a decision was taken to proceed with the erection of the full scale uranium extraction plants, there existed a critical shortage of uranium, and for strategic reasons it was essential that these plants be put into operation in as short a time as possible. From the time that the first leaching tests on the extraction of uranium from the residues from the gold mining operations on the Witwatersrand were undertaken in October, 1945, only 6 years elapsed before the first full scale extraction plant commenced operations in October, 1952(1).

2. In view of these circumstances it is not surprising that in the following ten years many modifications and new ideas have been developed and examined by the research laboratories of the mining industry in South Africa. Certain of these new processes have been introduced on the full scale plants, others have been tested on a pilot plant basis and have been shelved until an improvement in the marketing position makes their consideration worthwhile.

Outline of Original Process

3. The first uranium extraction plant at the West Rand Consolidated Mines incorporated the following main processing steps.

4. The crushed and ground ore in the form of a moist filter cake from the gold extraction plant, was made up into a slurry with water containing small amounts of acid. This slurry was agitated in Pachuca tanks and manganese dioxide (as oxidant) and sulphuric acid were added to dissolve the uranium. After filtration on rotary filters the leach liquor and filter washings were combined and after clarification were passed through columns containing anionic exchange resins. The uranyl sulphate complex anion was selectively absorbed by these resins, and was subsequently eluted with a nitric acid-ammonium nitrate solution to obtain a more concentrated solution of uranium containing only relatively small quantities of impurities such as iron, silica and aluminium. This concentrated solution, or eluate, was treated with lime to precipitate the iron as a hydrated oxide, and after filtration, with ammonia, to obtain ammonium diuranate of relatively high purity. This latter precipitate was separated by settling and filtration, and then calcined to obtain U_3O_8 as a final product.

Improvements in the Leaching Operation

Continuous Leaching

5. The first modification that took place in the original batch leaching operation was to convert the process to a continuous basis[2]. In the modified system the slurry was fed continuously to the first of a series of pachuca tanks in which the sulphuric acid was added. After passage through the first tank the slurry overflowed into the remaining nine or ten tanks in series. The addition of manganese dioxide was delayed until the third tank in series so that the decomposition of reducing constituents (such as metallic iron and sulphides in the ore) could take place before the addition of the oxidant. The total residence time of the slurry in the series of pachuca tanks had, of course, to be slightly longer than in the case of batch operation to make allowance for a slight amount of short circuiting. However, as a result of this modification a considerable improvement in the control of acid additions, the quantity of acid and manganese dioxide consumed, and the amount of manpower required for operation were effected. In addition such a system proved particularly convenient for the introduction of automatic control instruments such as mass-flowmeters, pH meters, redox-potential measuring systems etc., all of which assisted materially in providing uniform and consistent operation. At the same time steam injection was introduced to raise the temperature of the acidic slurry and this procedure increased significantly the percentage of uranium dissolved from the ore.

Ferric Leach

6. One of the major costs involved in the extraction of uranium is the cost of leaching reagents. Although sulphuric acid was produced locally from pyrite recovered from the ore by flotation, and there existed cheap and abundant supplies of manganese dioxide in South Africa, nevertheless the cost of these reagents represented a major proportion of the total processing cost. Accordingly considerable research was devoted towards reducing the cost of such reagents or alternatively attempting to replace these reagents with less costly ones. Perhaps the most successful modification has been installed at the uranium plants of the Virginia Gold Mining Company in the Orange Free State, and the Hartebeesfontein Gold Mining Company, Western Transvaal, and is referred to as the "ferric leach".[3][4]

7. In this process the oxidant used is the ferricion and acid is formed from sulphur dioxide produced by the oxidation of pyrite or burning of sulphur. Both the oxidant and the acid are produced by the well known auto-oxidation reaction:

$$4FeSO_4 + 2SO_2 + 2H_2O + 3O_2 \longrightarrow 2Fe_2(SO_4)_3 + 2H_2SO_4 \qquad (2)$$

8. In the plant operating at the Virginia Gold Mining Company's plant, the leaching reaction is conducted at temperatures of the order of 75-80°C and it has been shown that the dissolution of uranium has been increased by approximately 5% as compared with the conventional leach.

9. The hot leached slurry is passed through a heat exchanger and cooled to a temperature of approximately 40°C before being filtered and washed in the usual way. Approximately 40% of the filtrate obtained is used as a feed to the auto-oxidation section where the acid and ferric

concentrations are regenerated. The remaining 60% of the filtrate is diluted and treated by ion exchange in the conventional manner.

10. The solution to the auto-oxidation section is passed through the heat exchanger countercurrent to the outgoing leach slurry. The composition of the solution fed to the auto-oxidation plant is as follows:

Fe^{++} - 7.0 g/l
Fe^{+++} - 2.5 g/l
H_2SO_4 - 9.0 g/l

and after absorption of SO_2 and oxidation with atmospheric oxygen the following leach solution is obtained:

Fe^{++} - 0.1 g/l
Fe^{+++} - 9.5 g/l
H_2SO_4 - 12.5 g/l

11. One particular feature of the auto-oxidation plant used by the Virginia Gold Mining Company, is that the absorption of SO_2 is achieved by using a modified form of flotation cell, in which the SO_2 gas is finely dispersed in the solution by means of a high speed, mechanically agitated impellor.

12. The sulphur dioxide required for the auto-oxidation reaction is obtained from the standard contact sulphuric acid plant in which pyrite is used as the source of sulphur. Alternatively elemental sulphur can be burnt to form SO_2. The consumption of SO_2 is estimated to be approximately 4 - 6 lbs/ton and in addition 35 - 45 lbs of sulphuric acid per ton are used to dissolve ferric oxide residues from the sulphuric acid contact plant forming ferric sulphate which is added to the leach solution.

13. Many practical problems had to be overcome particularly as regards the choice of materials of construction. The full scale plant has however been operating for several years and significant savings in the leaching costs have been reported. The process is claimed to be fairly versatile and can be used to operate for maximum recovery of uranium or minimum cost of production.

Pressure Leaching

14. Another interesting approach to the problem of reducing leaching costs is the use of high temperatures and pressures for the 'in situ' oxidation of pyrite in the ore being treated(5). Under suitable conditions the pyrite is oxidized according to the following reaction:

$$2FeS_2 + H_2O + 7O_2 \longrightarrow 2Fe^{+++} + 4SO_4 + 2H^+$$

A portion of the ferric ion thus formed acts as oxidant for the dissolution of uranium, while the remaining portion is hydrolysed under the high temperature conditions to form an insoluble precipitate of hydrated ferric oxide

$$2Fe^{+++} + (n+3)H_2O \longrightarrow Fe_2O_3\,n\,H_2O + 6H^+$$

A large number of the uranium ores in South Africa contain a sufficient quantity of pyrite to permit sufficient acid to be formed for efficient leaching. Even in the case of those ores which contained little or no pyrite, the addition of pyrite appeared to be an interesting economic proposition.

15. This process, commonly referred to as "pressure leaching", was originally suggested by Forward[6] in Canada. Many workers have demonstrated the chemical feasibility of the process and similar test-work confirmed the feasibility for South African uranium ores. In fact, the preliminary laboratory scale test work demonstrated that there were several possible advantages to be derived from the process. Firstly the dissolution of uranium was significantly higher than in conventional processes, and secondly the leach solutions derived contained significantly less impurities than those obtained in the conventional process. However, at an early stage it was appreciated that the problems associated with the process were in the field of materials of construction and design and engineering for high pressure operation.

16. In order to assess these problems a pilot plant was erected and operated, and many interesting aspects of the process were brought to light. It was shown that:

(a) type 316 stainless steel (16-18% Cr, 10-14% Ni, 2-3% Mo) could be used as a material of construction for the high pressure autoclaves and for the heat exchangers.

(b) Heating of the slurry inside the autoclave could be obtained by steam injection without excessive dilution of the slurry.

(c) Compressed air could be used to provide a source of oxygen and could also be used to agitate the slurry in the autoclaves.

(d) Improved uranium extraction and the production of leach solutions containing much less impurities than in the conventional leach were also demonstrated.

17. It was also shown that the costs of the process were critically dependent on the quantity of air required to treat a given quantity of ore. Not only were the costs of compressing large volumes of air to the desired temperature and pressure a significant factor, but in addition the air passing through the autoclave became saturated with water vapour, and the latent heat of evaporation of this water constituted a serious source of heat losses to the system. The capital cost of a pressure leaching plant would almost certainly be greater than the cost of an equivalent conventional plant, but in general the results of the pilot plant experiments indicated that the saving in sulphuric acid and manganese dioxide more than compensated for the additional capital cost. However, at the stage that this work was conducted the outlook for long term future sales of uranium were not optimistic. It was not surprising, therefore, that no mining companies were prepared to consider abandoning existing plant in order to erect new plant at considerable additional capital cost. Nevertheless, it is considered that pressure leaching can seriously be considered for new projects.

18. Several other leaching processes have been examined, and mention should be made of an "autogenous in situ leaching technique". Such a process involves the formation of a slimes dam in such a way as to be permeable to water, and thus permit the ore containing sulphides to undergo a form of natural weathering. The dissolved oxygen in water circulated through the slimes dam material brings about oxidation of the pyrite, the formation of acid and dissolution of uranium. Although this is a slow process requiring many months for completion, it has obvious interest to certain South African mines where large quantities of uranium bearing ore have to be dumped until an increase in the demand for uranium occurs and their treatment becomes worthwhile.

Filtration of the Acid Leach Slurry

19. The filtration characteristics of the acid leached slurries are extremely unfavourable. In fact, the filtration problem was initially considered a most serious problem, until the discovery that small additions of animal glue improved filtration properties considerably. Continued research into the use of other flocculants or filter aids has brought about a considerable improvement in filtration procedures and costs, but nevertheless, this stage of the process still represents a considerable cost factor. Many ideas, such as countercurrent decantation, liquid-liquid extraction and resin in pulp processes have been suggested as a means of avoiding the filtration step. Countercurrent decantation as an alternative process was rejected at an early stage on account of practical difficulties and the extremely large plant required for handling the large tonnages of very finely ground ore slurries. Liquid-liquid extraction as applied to unfiltered leach slurries was rejected on the grounds of the excessively high solvent losses. However, a considerable amount of testwork was conducted on "resin in pump" processes with promising results. This work was described in detail by Read[7] and Carman[8] and only a brief description of the main features can be included in this paper.

20. The essential features of the resin in pump process are that the ion exchange resin beads are mixed with the acid leach slurry. After sufficient contact time to permit almost complete equilibrium to be established, the resin is separated from the slurry and the uranium recovered by elution with nitric acid as in the conventional process. The particular features of the pilot plant described by Read are:

(a) A countercurrent system in which the resin and slurry are mixed in series of pachuca type agitators, and after separation the resin and slurry move in opposite directions to the next mixing tanks so as to provide a countercurrent multistage operation. In this way the overall recovery of uranium can be well in excess of 99%.

(b) An ingenious inclined separator which permits rapid and efficient separation of the resin beads and the slurry with a very small entrainment loss.

21. In the operation of the pilot plant, the resin losses were exceedingly small, and the recovery of uranium from solution was at least as good as that obtained by the conventional process. A considerable saving of $0.375/ton treated is claimed as a result of reduced capital, increased uranium recovery and elimination of filtration costs.

Modifications to the Conventional Ion Exchange Process

22. When the first uranium extraction plant commenced operation, one of the most serious problems encountered was the operation of the ion exchange process. These difficulties were due almost entirely to the presence of "poisons" in the leach liquors. These poisons were substances which were preferentially and irreversibly absorbed onto the resins and resulted in a rather rapid deterioration in the capacity of the resins for uranium. The poisons causing most of the deterioration were the polythionate and cobalt/Cyanide complexions. However, as a result of a considerable amount of intensive research this problem has now largely been overcome, and contrary to many opinions, the ion exchange process has been developed into an extremely efficient processing technique.

23. The difficulties due to polythionate poisons were overcome by careful control of the leaching operation to avoid conditions which favour the formation of these compounds, and also by the use of higher concentrations of nitric acid in the eluting solutions, so as to bring about oxidation of the polythionates absorbed on the resin[9][10]. In the case of the cobalt/cyanide complex poisons no simple solution to the problems was forthcoming other than to take every possible precaution to avoid their formation. More recently, however, Nugent[11] and other workers have developed the regeneration procedures to a point where they are claimed to represent a practical and economic method of regenerating cobalt poisoned resins. A regeneration pilot plant was operated at the West Rand Consolidated uranium plant and a full scale plant has now been installed.

24. Two processes are available to remove cobalt/cyanide complexes from the resin. In the first process use is made of a hot 8% (W/V) ammonium thiocyanate solution to convert the cobalt cyanide complex to a thiocyanate complex. This complex can then be eluted from the resin with 7.5% sulphuric acid at near boiling temperatures. Over 90% of the cobalt can be removed by this treatment and only a very slight decrease in the total capacity of the resin after this severe treatment was observed. In the second process, use is made of a hot sodium nitrite solution as a regenerant. The exact mechanism of the regeneration reaction is not fully understood but it was indicated that the reaction was reversible so that a countercurrent multistage process had to be used. One particular advantage of this regeneration procedure was that cobalt could be recovered from the effluent solutions by the addition of copper sulphate when an insoluble cobalt precipitate was formed. After precipitation of the cobalt and filtration, the filtrate, which contained appreciable quantities of sodium nitrite could be recycled and reused. Particular attention has been devoted to the effect of these regeneration procedures on the subsequent performance of the resins in extracting uranium from leach liquors, and there is little evidence to indicate any serious increase in the normal rate of deterioration. A careful assessment has been made of the economics of resin regeneration, and Nugent[11] estimates that in the case of the West Rand Consolidated Plant a saving of approximately £37,000 per annum can be effected by the use of regenerated resin as compared with the purchase of new resin.

Economic Assessment of the Ion Exchange Process

25. Detailed attention has been given to the assessment of the cost

of the ion exchange process particularly as regards the choice of the most suitable resin to be used and the optimum regeneration and replacement frequencies[12]. An attempt has been made to incorporate all the factors that influence the cost of ion exchange processing into one general mathematical formula which can then be used to ascertain the relative advantages of different resins and the most economical operating procedure for any given conditions. Attention to these aspects has resulted in much improved operation and a significant reduction in the processing costs.

Liquid-Liquid Extraction

26. A great deal of attention has been devoted in the U.S.A. and Canada to the use of liquid-liquid extraction techniques as an alternative to ion exchange for the recovery of uranium from leach liquors. Comparatively little attention has, until recently, been devoted to this technique in South Africa.

27. This state of affairs is undoubtedly due to two factors:

(a) the efficiency and extremely low cost of the ion exchange process once the preliminary poisoning difficulties had been overcome.

(b) the low concentrations of uranium in the South African leach liquors and the difficulty in obtaining an organic solvent capable of efficiently extraction uranium from such dilute leach solutions.

28. However, recently a systematic survey of a large number of solvents has been undertaken by Lloyd[13], and the most promising has been used to make a preliminary estimate of the processing cost of a liquid-liquid extraction process as compared with ion exchange. The interesting conclusion was reached that the liquid-liquid extraction process could successfully compete in terms of processing cost and efficiency, with the ion exchange process. The solvent used in making this comparison was a tertiary amine, "Amberlite XA 204" and the most suitable procedure suggested by Lloyd included:

(a) an extraction step using a 0.1 M solution of the amine in a kerosene diluent. Four mixer settler stages, operating in a continuous countercurrent process, gave an extraction of uranium of over 99.8% from the pregnant solution.

(b) a stripping step using a 1.0 M aqueous solution of sodium chloride as the stripping solution. The stripped solvent was recycled directly to the extraction section.

29. Lloyd estimates that on the basis of laboratory scale tests it should be possible to replace the existing ion exchange process by the proposed liquid extraction cost, repay any capital expenditure involved in eight years and result in an additional saving of approximately £42,000 per annum.

Precipitation Process

30. No fundamental changes have been made to the precipitation processes for recovering uranium as an ammonium diuranate from the strong eluates. Nevertheless, careful attention to details such as the control of the

precipitation conditions and the concentration of sulphate ions in the eluate have brought about a steady improvement in the purity of the final product. When the first plants commenced operations the grade of the final product was approximately 70%. At present, the average grade of the products from all the existing producers is over 90%.

Comparison of Different Processes

31. It is of interest and value to speculate what would be the processes used if a new uranium plant were to be designed. This is perhaps an academic speculation since in view of the depressed marketing conditions, it is unlikely that any new producers would commence operations at the present time, and it is more than probable that many unforeseen improvements and modifications will be suggested before new plants can be justified. Nevertheless, a speculation of this kind is of value in providing a means of assessing the relative merits of the various new processes that have been suggested.

32. To some extent the choice of processes would depend on whether the plant as a whole is to be designed for maximum recovery or minimum cost. At the present time it is likely that a process would be chosen to produce uranium at minimum cost. However in the post 1970 periods it is not unlikely that uranium will be in short supply, and emphasis may once again be placed on maximum recovery.

33. Thus in the immediate future it is probable that serious consideration would be given to the use of flotation as a preliminary concentration step prior to chemical treatment. Although no major advance has been made in the flotation of uraninite from South African ores, and the recovery can be expected to be low, the degree of concentration that could be achieved would result in a very much smaller quantity of material to be handled at all stages which in turn would probably represent a most significant reduction in cost. In dealing with a pyritic ore, the relatively large quantity of pyrite in the concentrate would suggest that the pressure leaching process would be the most attractive leaching procedure. In view of the relatively small quantities of material to be handled, and the fact that small losses of uranium in the filter cake would be of little significance it is probable that the standard filtration procedure would be adopted. The filtration properties of the pressure leached slurry are considerably better than the normally leached slurries and it may even be possible to dispense with the addition of filter aids, which would also represent a saving in cost.

34. The higher uranium concentration in the leach solutions would probably favour the use of a liquid-liquid extraction procedure for concentration and recovery of uranium.

35. In the case of a plant designed to achieve maximum possible recovery of uranium, a completely different sequence of processes might be anticipated. In this instance direct leaching of the ore would certainly provide maximum possible recovery. It is quite possible that the ferric leach system operating at high temperature would be selected as the most economic means of obtaining maximum recovery.

36. For treating the acid leached slurry the promising result obtained by the resin-in-pulp pilot plant suggest that this process would not only result in decreased capital expenditure, but would also obviate the 1-2% soluble losses of uranium in the filter cake. The elution of the resins

and the precipitation of the uranium oxide is likely to follow the pattern as established at present. With the above sequence of processes it is possible that well above 95% uranium recovery could be achieved at a lower cost than that pertaining in the present plants.

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SUMMARY

RECENT ADVANCES IN THE EXTRACTION OF URANIUM FROM SOUTH AFRICAN ORES

R.W. Robinson
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A Review is given of the developments that have occurred since the first South African uranium extraction plant commenced operation in October, 1962. Consideration is given to the following aspects of the uranium extraction process. (a) Leaching. A brief description is given of the "ferric leach" process as installed by the Virginia Gold Mining Co., and the investigations on the application of pressure leaching to South African Ores. (b) Filtration. Consideration is given to the various ways of avoiding this step in the processing and particular attention is given to the resin-in-pulp process for the treatment of unfiltered slurries. (c) Treatment of leach solutions containing uranium. A description is given on the work conducted on the regeneration of poisoned ion exchange resins and the assessment of the liquid-liquid extraction process as an alternative to the ion exchange process.

Not all the advances have been applied on a full scale in South Africa since additional capital would have to be expended in abandoning old plant and erecting new plant, and it is doubtful whether such expenditure could be justified in view of the limited market for uranium existing at present.

However, for countries contemplating the establishment of new uranium extraction plants, it is probable that the developments indicated would be of interest. Consideration is therefore given to the economic, marketing and other factors which could influence the choice of the particular processes to be used for uranium extraction.

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